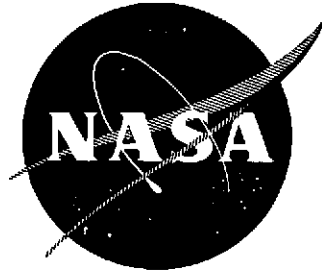


P
24P

NASA CR-121267



FURTHER DEVELOPMENT OF CHEMICAL VAPOR DEPOSITION PROCESS FOR PRODUCTION OF LARGE-DIAMETER CARBON-BASE MONOFILAMENTS

by R. L. Hough and R. D. Richmond

NASA-CR-121267) FURTHER DEVELOPMENT OF
CHEMICAL VAPOR DEPOSITION PROCESS FOR
PRODUCTION OF LARGE DIAMETER CARBON-BASE
MONOFILAMENTS Contractor Report. May
(Hough Lab.)

SCL 13H

G3/15

N74-20068

Unclas
33454

HOUGH LABORATORY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center

Contract NAS 3-15837

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
US Department of Commerce
Springfield, VA. 22151

1. Report No. NASA CR-121267	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle FURTHER DEVELOPMENT OF CHEMICAL VAPOR DEPOSITION PROCESS FOR PRODUCTION OF LARGE-DIAMETER CARBON-BASE MONOFILAMENTS		5. Report Date February 1974
		6. Performing Organization Code
7. Author(s) R. L. Hough R. D. Richmond		8. Performing Organization Report No.
9. Performing Organization Name and Address Hough Laboratory		10. Work Unit No.
		11. Contract or Grant No. NAS3-15837
12. Sponsoring Agency Name and Address NATIONAL AERONAUTICS & SPACE ADMINISTRATION Washington, D. C. 20546		13. Type of Report and Period Covered Contractor Report May 1972 to May 1973
		14. Sponsoring Agency Code
15. Supplementary Notes Project Manager, David L. McDanels, Materials & Structures Division, NASA Lewis Research Center, Cleveland, Ohio		
16. Abstract <p>Research was conducted to further develop large diameter carbon-base monofilament in the 50 micron to 250 micron (2.0 mil to 10 mil) diameter range using the chemical vapor deposition process. The objective of this program was to determine the critical process variables which control monofilament strength, monofilament modulus, and monofilament diameter. It was confirmed that wide scatter in the carbon substrate strength is primarily responsible for the scatter in the monofilament strength. It was also shown through etching experiments that defective substrate surface conditions which can induce low strength nodular growth in the monofilament layers are best controlled by processing improvements during the synthesis of the substrate. Modulus was found to be linearly proportional to monofilament boron content. Filament modulus was increased to above 27.8MN/cm² (40 × 10⁶ psi) but only by a considerable increase in monofilament boron content to 60 wt. % or more. Monofilament diameter depended upon dwell time in the synthesis apparatus.</p> <p>A monofilament was prepared using these findings which had the combined properties of a mean U.T.S. of 398,000 N/cm² (568,000 psi), a modulus of 18.9 MN/cm² (24 × 10⁶ psi), and a diameter of 145μ (5.71 mils). Highest measured strength for this fiber was 451,000 N/cm² (645,000 psi).</p>		
17. Key Words (Suggested by Author(s)) Carbon, Monofilaments, Composites, Fibers Chemical Vapor Deposition, Reinforcement		18. Distribution Statement Unclassified - unlimited
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. N

* For sale by the National Technical Information Service, Springfield, Virginia 22151

TABLE OF CONTENTS

SECTION	PAGE
Summary	1
I. Introduction	2
II. Materials, Apparatus & Procedure	9
A. Materials	9
B. Apparatus	10
C. CVD Process Procedure	11
D. Monofilament Evaluation	14
III. Results & Discussion	17
A. General	17
B. Effect of Process Variables on Tensile Strength	18
C. Effect of Process Parameters on Modulus of Elasticity	29
D. Effects of Process Parameters on Diameter	32
E. Summary of Effect of Process Variables on Properties	33
F. Cost Reduction, Quality Control and Production Capability	35
IV. Conclusions	36
References	38
Appendix	41

Preceding page blank

LIST OF ILLUSTRATIONS

Figure	Title	Page
1	Strength vs. Diameter - Selected Carbon Monofilaments - - -	3
2	Ultimate Tensile Strength vs. Elastic Modulus for Fibers and Yarns- - - - -	7
3	Production Line Schematic - - - - -	12
4	Production Apparatus- - - - -	13
5	Effect of Boron Content on Fiber Properties - - - - -	19
6	Substrate Strength Variations with Length - - - - -	22
7	Monofilament Boron Content vs. Modulus- - - - -	31

SUMMARY

A research program was conducted to further develop the chemical vapor deposition process for the production of large diameter carbon-base monofilaments. Target property goals were an elastic modulus of 40.7 MN/cm^2 ($58 \times 10^6 \text{ psi}$), a tensile strength of $406,000 \text{ N/cm}^2$ ($580,000 \text{ psi}$), and a diameter range of 75 to 250μ (3 to 10 mils). This work extended to include independent effects of process variables on filament strength, filament modulus, and filament diameter. It was intended to isolate those overriding variables which control fiber properties and which had not been defined in previous research programs.

Previous contracts showed that large diameter carbon-based monofilaments can be made but that problems existed which prevented the successful attainment of a viable production process. These problems include scatter in strength, limitations in diameter and modulus, and substrate surface defects.

This program was organized on the basis of the filament property variables of strength, modulus, and diameter. Each filament property was studied to determine the critical variable or variables which have the prime effect on that fiber property and which have promoted successful and economical fiber production.

The findings of this program confirmed that wide scatter in the carbon substrate strength is primarily responsible for the scatter in the monofilament strength. These findings also show that defective substrate surface conditions which can induce low strength nodular growth in the monofilament layers are best controlled by processing improvements during the synthesis of the substrate, and that the monofilament strength tends to be relatively insensitive to process variables. Modulus was found to be dependent on boron content. The results further show that filament modulus can be increased to above 27.6 MN/cm^2 ($40 \times 10^6 \text{ psi}$) but only by a considerable increase in boron content to $60 \text{ wt. } \%$ or more. Filament diameter depended, as might be predicted, upon the dwell time in the synthesis apparatus.

A filament was prepared using these findings which had the combined properties of a mean U.T.S. of $398,000 \text{ N/cm}^2$ ($568,000 \text{ psi}$), a modulus of 18.9 MN/cm^2 ($27 \times 10^6 \text{ psi}$), and a diameter of 145μ (5.71 mils). Highest measured strength for this fiber was $45,000 \text{ N/cm}^2$ ($645,000 \text{ psi}$).

I. INTRODUCTION

As the performance requirements for aerospace propulsion systems increase, a corresponding need develops for increasing materials capabilities. One area of materials development involves improving the high temperature capabilities of metals and ceramics which are intended for use in turbine engines. Fibrous reinforcements which both strengthen and stiffen metals and ceramics are being considered for achieving these desired improvements. Reinforcements based on carbon are particularly attractive in this respect because of the potentially high strength-to-weight ratio.

Carbon in fibrous form has been known for many years. It was first reported in 1890 by Paul and Leon Schestzenberger who obtained a bulky mass of fibers by thermal decomposition of carbon containing gases (Reference 1) heated in a porcelain tube. In 1903 Constant and Pelabon (Reference 2) found graphite wool formations in coke ovens near the hot gas exits. Such filamentary structures were considered as mere scientific curiosities. In 1960 however, Bacon (Reference 3) reported the successful experimental demonstration of the strength potential inherent in the carbon structure (excluding the cubic form). Bacon grew graphite whiskers under near-equilibrium conditions in a D.C. electric arc. The whisker diameters ranged from 0.5μ to 5.0μ (0.02 to 0.2 mils) with basal planes parallel to the fiber axis. Ultimate tensile strength of 1.9 MN/cm^2 ($2.8 \times 10^6 \text{ psi}$) and a modulus of nearly 96.4 MN/cm^2 ($140 \times 10^6 \text{ psi}$) were measured for the smallest whiskers or for ribbon shaped whiskers where a large gripping surface-to-volume ratio minimized basal plane slippage. That such slippage is a limiting factor in actual measured strength is shown in Figure 1, where the point A denotes a U.T.S. value for a 5.0μ (0.2 mil) sublimed whisker (Reference 4).

The highly anisotropic nature of the near perfect graphite (i.e., the very high strength in the "a" direction and weak delamination resistance in the "c" direction) limits the practical fiber strength for larger diameter monofilaments of perfectly ordered graphite. Less highly ordered graphitic monofilaments may therefore exhibit higher measured strength because of intrinsic locking mechanisms which inhibit basal plane slippage.

M. Hillert and N. Lange grew polycrystalline graphite filaments by pyrolysis of hydrocarbons at 1000°C (1832°F) in a silica tube furnace. Bacon

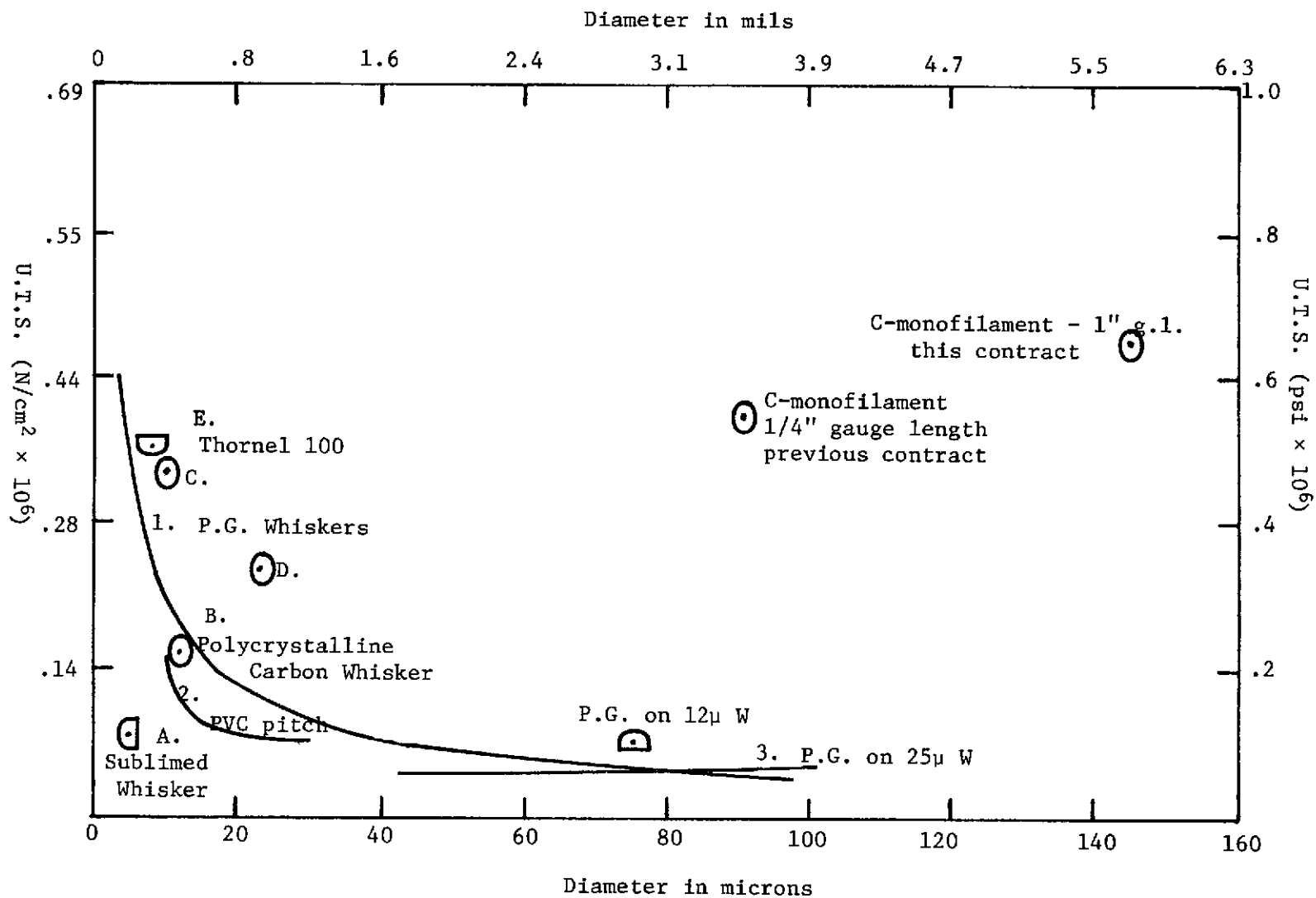


Figure 1 - Strength vs. Diameter - Selected Carbon Monofilaments

(Reference 3) measured the tensile strength of these fibers and found a 12 μ (0.5 mil) diameter filament with U.T.S. of 152,000 N/cm² (220,000 psi). (See point B of Figure 1.)

R. G. Bourdeau (References 5 and 6) synthesized pyrolytic graphite whiskers over a wide range of diameters. These whiskers grew in the temperature range of from 1000°C to 1400°C (1832°F to 2552°F). Tensile strength vs. diameter is shown as curve no. 1 in Figure 1. Points C and D represent isolated, yet extremely interesting, values also measured by Bourdeau. The fiber with strength displayed at point C is found to have a modulus of 63.7 MN/cm² (92.4 $\times 10^6$ psi). The point D represents the highest combined values of diameter, strength and modulus measured in one fiber. Unfortunately, the work of Bourdeau was never published in the open literature, nor was additional research performed. The synthesis parameters are complex, and years of research may very well be required before pyrolytic graphite whiskers are obtained having reproducible properties (Reference 7).

Other methods for forming filamentous carbon include the stress-graphitization of rayon or other organic yarns. The result is a fine-grained, relatively disordered microstructure which never fully develops into a three-dimensional ordering. Therefore, there is no tendency for delamination. Point E in Figure 1 represents a single fiber extracted from stress-graphitized carbonized rayon yarn as measured by Bacon (Reference 8). There is apparently no information available on strength vs. diameter for larger diameter stress-graphitized carbon monofilament.

Otani (Reference 9) reports tensile strengths of nominally 68,900 N/cm² (100,000 psi) for 25 μ (1.0 mil) carbon monofilament derived by pyrolysis of polyvinyl chloride pitch. His data are displayed as curve no. 2 in Figure 1. Similar strength is reported by the Great Lakes Carbon Corporation for glassy carbon monofilament having a highly cross-linked structure (Reference 10). Strengths of from below 27,600 N/cm² (40,000 psi) to nearly 103,500 N/cm² (150,000 psi) have been measured on these glassy 25 μ (1.0 mil) diameter monofilaments in our laboratory.

The application of strain annealing to pyrolytic graphite will improve dimensional stability and improve physical properties unless the pyrolytic graphite structure is relatively defective. Thus, Stover and Berning (Reference 11) have demonstrated increased tensile strength for bulk strain-annealed

pyrolytic graphite at both room and elevated temperatures. Because of time limitations, Bourdeau (Reference 7) did not investigate the effects of strain-annealing on the pyrolytic graphite whisker material beyond a nominal 5% elongation. Regrettably, this is not enough to produce any significant effects on strength and modulus.

An internal program in the Plastics Branch, Nonmetallic Materials Division of the Air Force Materials Laboratory from March 1962 to July 1963 demonstrated the feasibility of forming continuous pyrolytic graphite filaments on a tungsten substrate in lengths of 793 meters (2600 feet) (Reference 12). Fiber strength vs diameter on 25 μ (1.0 mil) diameter tungsten is shown by curve no. 3 in Figure 1 (Reference 13). It was recognized that the 25 μ (1.0 mil) diameter tungsten substrate was not ideal because of the weight penalty attending the high density. Also, Kirkendall voids formed between the core and the graphite layer due to the rapid diffusion of carbon into the tungsten lattice. Thus, the core tended to contribute nothing toward mechanical properties. Twelve microns (0.5 mil) diameter tungsten was therefore considered as it would occupy less volume in the fiber and present a smaller diffusion sink. Before large diameter graphite fibers could be demonstrated utilizing the 12 μ (0.5 mil) tungsten, internal research programs were redirected toward the then emerging boron fiber program. Thus, curve no. 3 in Figure 1 represents maximum strength/diameter obtained (Reference 13) before project termination.

Under company-sponsored research, Texaco Experiment Inc. continued development of carbon monofilament deposited on 12 μ (0.5 mil) tungsten. Seventy-six micron (3.0 mil) diameter fiber was formed with a modulus of 12.4 MN/cm² (18.0×10^6 psi) and a tensile strength of about 68,000 N/cm² (100,000 psi) (Reference 14). Research and development of fibers and composites was abandoned at Texaco beginning in 1968 (Reference 15).

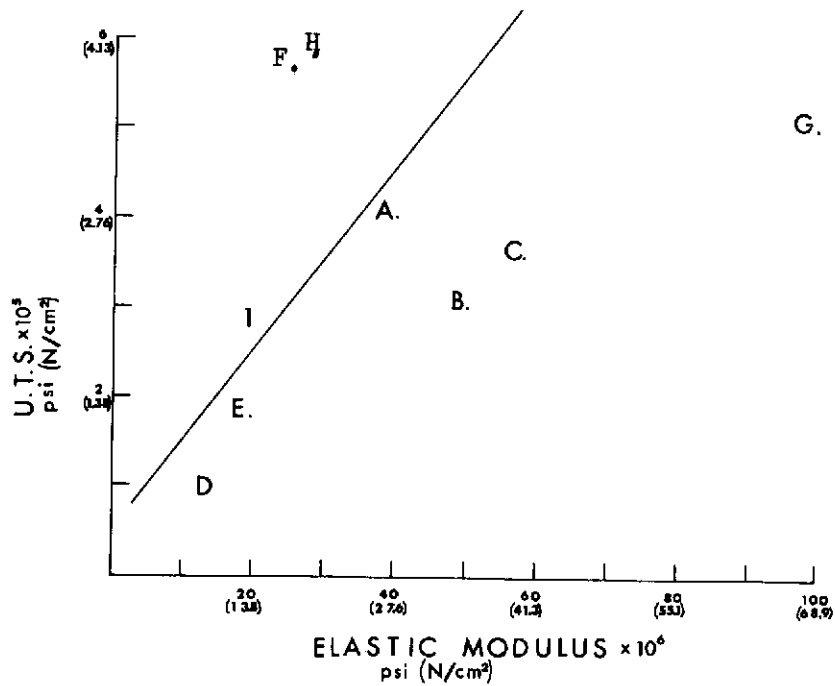
Carbon yarns have been commercially available since 1959. More recently, "graphite" yarns have been available. Crystallite reorientation of these yarns will increase the elastic modulus and the tensile strength. Similar reorientation by mechanical drawing produces an increased elastic modulus and tensile strength for thermoplastic polymers. Carbon yarns can be stretched at least 50 to 60 per cent under proper conditions of time, temperature, and

applied force. The elastic modulus may increase from 3.4 MN/cm^2 - 5.5 MN/cm^2 (five to eight million psi) to nearly 68.9 MN/cm^2 (100 million psi). Tensile strength may go from $68,900 \text{ N/cm}^2$ (100,000 psi) to nearly $275,600 \text{ N/cm}^2$ (400,000 psi). The hot stretched carbon yarn is usually composed of discrete crystallites joined by some amorphous carbon. The precursor polymer and the effect of such pyrolysis conditions as time, temperature, and atmosphere have a pronounced effect on final properties. The crystal orientation tends to approach the more perfect ordering found in pyrolytic graphite whiskers.

Because of the method of formation, graphite fibers derived by pyrolysis of polymeric precursors will contain some defects and voids which are virtually impossible to eliminate. That this is indeed the case is documented by the fact that, although high modulus values have been observed, a corresponding increase in tensile strength has not been noted. Therefore, modulus vs strength values (points A, B & C, Figure 2) plotted for these fibers fall below the plotted curve for pyrolytic graphite whiskers. The pyrolytic graphite whiskers themselves contain some voids and defects since measured values for the perfect graphite whisker crystals synthesized by Bacon lie above this curve.

In small diameter fiber ($<10\mu$), the defect mechanism does not appear to be strength limiting. However, as filament diameter increases, the probability increases that voids and defects will arise during char formation. This is particularly true since gaseous products stand less chance of escaping from the interior of a large mass. On the other hand, voids and defects, while probable, can be more easily controlled in pyrolytic deposits.

Currently, two approaches are being investigated to develop large diameter carbon-based monofilaments. One approach is the impregnation of a bundle of carbon fibers (yarn or tow) or their precursors with a resin and, after curing, developing the composite fiber by controlled pyrolysis. This approach has been studied by NASA - Lewis sponsored work in the United States by Quackenbush (Reference 16), Bradshaw, and others (Reference 17), and also more recently in England by Cooper and co-workers (Reference 18). Generally, such composite fibers contain cracks and voids within the matrix. A potentially attractive approach mentioned by both Cooper and Bradshaw involves the co-pyrolysis of the matrix material and the fiber precursor. This method is believed to favor an improved fiber-matrix interface, while reducing or elimin-



- A. Courtaulds (HT) yarn (Mfr. spec.)
- B. Thornel 50 yarn (Mfr. spec.)
- C. Courtaulds (HM) yarn (Mfr. spec.)
- D. Pyrolytic graphite on tungsten (Ref. 12)
- E. Pyrolytic graphite on carbon (Ref. 19)
- F. Carbon alloy monofilament (Ref. 19)
- G. Thornel 100 yarn
- H. Carbon alloy monofilament (Ref. 20)
- I. Pyrolytic Graphite Whiskers (Ref. 6)

Figure 2 - Ultimate Tensile Strength vs. Elastic Modulus
for Fibers and Yarns

ating pyrolysis cracks. The highest tensile strength reported by Bradshaw (Reference 17) is $134,000 \text{ N/cm}^2$ (195,000 psi) using Thorne 75 as the carbon fiber reinforcement.

The second approach studied under NASA - Lewis sponsored work is to synthesize continuous large diameter filaments by chemical vapor deposition. This approach involves the decomposition of a gaseous atmosphere at or near a substrate to achieve a solid deposit. This synthesis normally begins with a core material in the form of a tiny continuous fiber, such as fine tungsten wire or carbon fiber. This core is used as a nucleus upon which the required filament material is vapor deposited.

The feasibility of synthesizing high strength, continuous carbon-base monofilament by chemical vapor deposition upon a carbon substrate was established under a previous NASA contract (Reference 19). These filaments were synthesized by chemical vapor deposition utilizing a "hot wire" apparatus. A 33μ (1.3 mil) carbon substrate was drawn through the apparatus at a rate of $3.8 \times 10^{-3} \text{ mps}$ (0.75 fpm). Monofilaments in lengths of up to 61 meters (200 feet) were synthesized in the 76 to 102μ (3.0 to 4.0 mil) diameter range having measured ultimate tensile strength up to $372,000 \text{ N/cm}^2$ (541,000 psi) and an elastic modulus of 19.1 MN/cm^2 ($27.7 \times 10^6 \text{ psi}$). The high strength filaments were synthesized by alloying the carbon with a nominal 25 to 35 mole per cent elemental boron. Without the addition of boron, the carbon fibers were relatively weak, having maximum measured tensile strengths of ca. $110,000 \text{ N/cm}^2$ (160,000 psi) for fibers of nominal 70μ (2.75 mil) diameter.

A subsequent NASA contract (Reference 20) was directed toward studying variables which might lead to reduced fiber property scatter, higher fiber synthesis rate, and larger fiber diameter. Such variables included plating gas control and distribution, CVD reactor power control, and fiber synthesis velocity. An additional variable found was the condition of the carbon substrate surface, as received from the supplier.

Fibers were synthesized having experimental U.T.S. values ranging up to $381,000 \text{ N/cm}^2$ (552,000 psi); modulus values to nearly 21 MN/cm^2 ($30 \times 10^6 \text{ psi}$); and diameters to nearly 152μ (6 mils). However, synthesis parameters were not established which could produce these properties simultaneously in the same fiber. Synthesis speeds reached $12.9 \times 10^{-3} \text{ mps}$ (2.54 fpm) for 84μ

(3.3 mil) diameter fiber. This represents a 338% increase over synthesis speed achieved under the previous contractual effort. It was discovered that modulus is dependent in an undefined manner on boron content.

The objective of the work described herein was to further develop the chemical vapor deposition process for the production of large diameter carbon base monofilaments. Target property goals were an elastic modulus of 40.7 MN/cm² (58×10^6 psi), a tensile strength of 406,000 N/cm² (580,000 psi), and a diameter range of 75 to 250 μ (3 to 10 mils). This work extended to include independent effects of process variables on filament strength, filament modulus, and filament diameter. It was intended to isolate those overriding variables which control fiber properties and which had not been defined in the previous efforts.

II. MATERIALS, APPARATUS & PROCEDURE

A. MATERIALS

1. Substrate

The substrate used is carbon monofilament obtained from the Great Lakes Carbon Corporation. This material has the following vendor stated properties:

Diameter	33 μ (1.3 mils)
Tensile Strength	68,950 N/cm ² (100,000 psi)
Modulus	3.3 - 4.1 MN/cm ² ($4.8 - 6.0 \times 10^6$ psi)
Density	1.61 - 1.65 g/cc

The substrate was initially screened by removing several turns from the supply spool and microscopically examining a randomly selected 2.54 cm (1 inch) length for surface flaws. These flaws included spheres, grooves, pits, etc., and are described more fully in Reference 20. Generally, one defect per 1/d ratio of 10 or higher was not considered excessive. Continued processing improvements at Great Lakes are markedly reducing the frequency of such flaws.

In order to more clearly understand the dependence of the final mechanical properties, strength and modulus, on the quality of the substrate, tests

were made on various substrate lots and the results compared to the properties of the resultant fiber. Variations in such properties as resistivity, strain to failure, U.T.S., and modulus were studied for various substrate lots.

2. Reactant Gas

During this contract, different boron and carbon source combinations and various ratios of each combination were studied.

Boron Trichloride - Methane - Illustrated in Table I are experiments using BCl_3 and methane. Also shown (experiments 273 & 274) is work using BCl_3 and ethylene. The boron-to-carbon ratios ranged from 3.3:1 to 1:2.4.

Diborane - Ethylene - Table II shows the experiments performed using the diborane - ethylene system. The boron-to-carbon ratio ranged from 1:102 to 1:40.

Triethylborane - Ethylene - Displayed in Table III are experiments performed with the borane and ethylene. Here boron-to-carbon ratios were from 1:22 to 1:52.

Ternary Si-B-C - A fourth system that was studied briefly was a combination Si-B-C system. In 1965, Marquardt Corporation produced CVD Si-B-C material on 13μ (0.5 mil) tungsten wire (Reference 21). This fiber demonstrated strengths exceeding $270,000 \text{ N/cm}^2$ (400,000 psi) with elastic modulus in excess of $48 \times 10^6 \text{ N/cm}^2$ (70×10^6 psi).

In attempting to produce the same type of material on 33μ (1.3 mil) carbon substrate, we used this reactive gas mixture:

Trichlorosilane, boron trichloride, methane

In this mixture, we used a ratio of 50:1:50, silicon to boron to carbon. The results are illustrated in Table IV. This gas system was not a duplicate of that used by Marquardt; and the data is not sufficient to determine if such a system does work. However, no significant data was obtained to justify a further expenditure of time and effort.

B. APPARATUS

The processing apparatus utilized in this development program is essen-

tially a series of long cylinders (Figures 3 & 4) through which the substance material is drawn by means of a motor driven take-up spool. The number of cells was varied in this program between three and five. Cell lengths were as follows: cleaning cell, 33 cm (13 in); first plating cell, 95 cm (37 in); second, 117 cm (46 in); third, 115 cm (45.5 in); fourth, 112 cm (44 in). A simple braking device on the supply spool provides proper substrate tension. The ends of each cylinder are sealed by mercury filled capillaries which allow passage of the fiber while preventing interchange of plating gases and air. Hot zone lengths are nominally 5 cm (2 in) less than stated cell lengths. Fiber temperature measured approximately 2.54 cm (1 in) after the fiber entered the cells.

The chemical vapor deposition reaction is initiated by the "hot wire" method. The substrate is drawn through the mercury seals which also serve as electrical contacts. It is then resistively heated to the required deposition temperature by application of controlled electrical power. The main factors which control the deposition process include:

1. Plating gas distribution and control;
2. CVD reactor power for controlling temperature; and
3. Fiber synthesis velocity control.

C. CVD PROCESS PROCEDURE

1. Plating Gas Distribution and Control

Laminar flow elements with a manufacturers' stated accuracy of 0.5% of full scale are used without further calibration to monitor gas flow. These elements have ranges of 1.0 to 10.0 lpm referenced to air. We employed the direct and reliable procedure of utilizing differential manometers, readable to 0.025 cm (0.01 inches). Gases can be mixed and distributed to various stages of the synthesis line under close monitoring. Thus, under controlled variations, the relationship of flow rate and gas composition on fiber properties can be established. Nominally 450 - 500 ml/min argon, 300 - 400 ml/min hydrogen, 80 - 110 ml/min C_2H_4 , and 3 - 5 ml/min $(C_2H_5)_3B$ flowed through each cell. Tables V, VI, and VII illustrate the heating currents and rela-

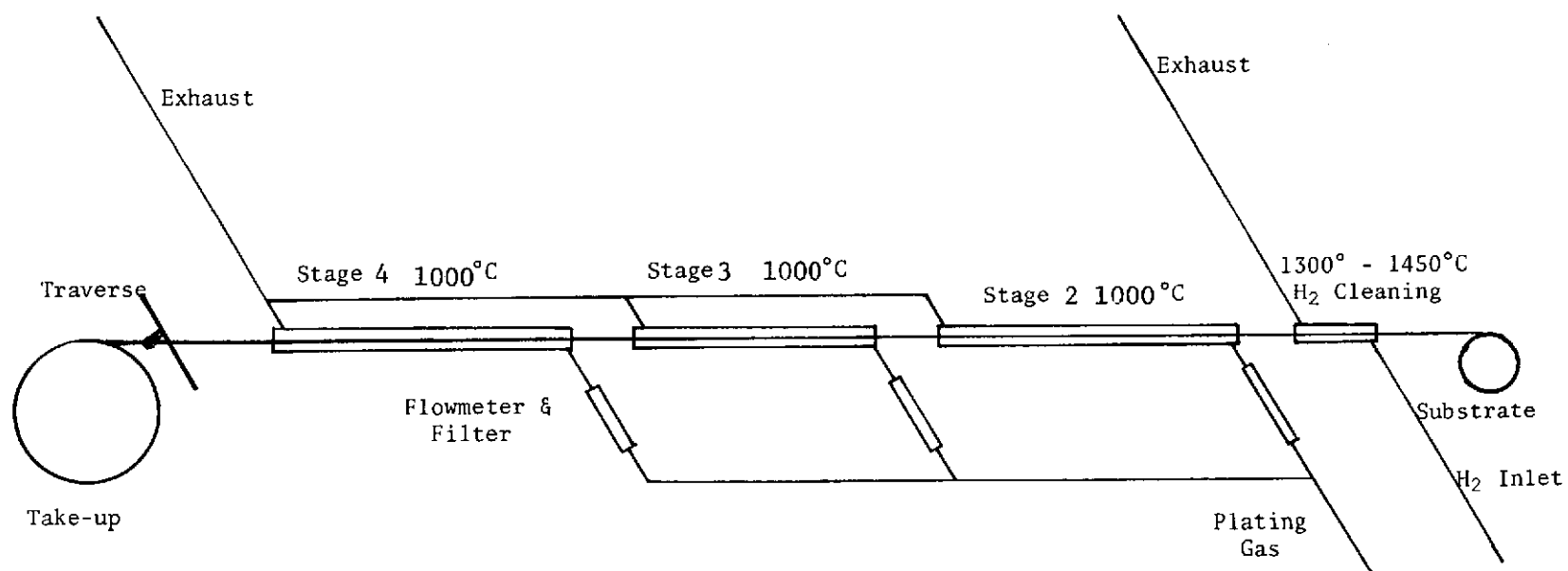


Figure 3 - Production Line Schematic

This page is reproduced at the back of the report by a different reproduction method to provide better detail.

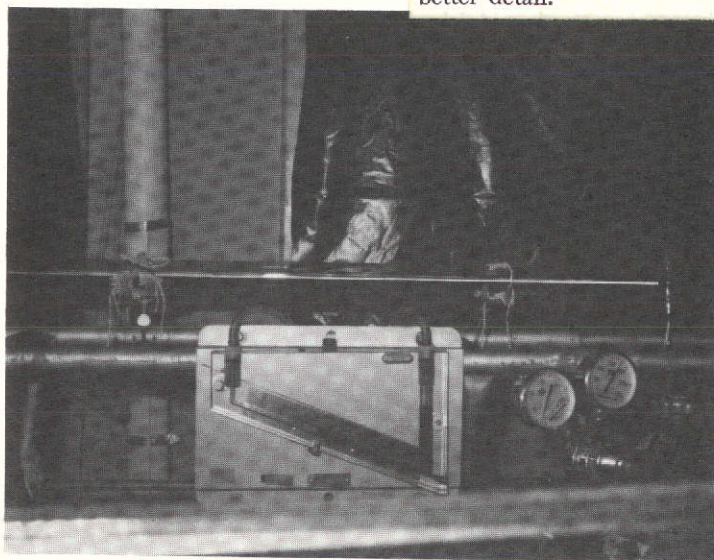


Figure 4 - Production Apparatus

tive gas flows in the plating cells during the experiments in Tables I, II, and III respectively.

2. CVD Reactor Power for Controlling Temperature

CVD reactor power control is obtained to within ± 1 ma of the setting range 100 - 150 ma. The power controls use magnetic amplifiers which operate the primaries of high voltage (2000 - 2500 volts) transformers feeding rectifier and filter banks. A solid state feedback loop senses current output and adjusts the current level in the control circuit of the magnetic amplifiers to compensate for any spurious drifting effects.

Substrate temperature is measured using a micro-optical pyrometer manufactured by the Pyrometer Instrument Company. Temperatures were measured 2.54 cm (1 in) after the fiber entered the cells. Measurement accuracy and repeatability are conservatively estimated at $\pm 50^\circ\text{C}$ or less. Temperature is spot-checked and not measured regularly. One reason for this is that the plating cell walls become clouded during prolonged experimentation. Accurate visual measurements then become impossible. A second reason is that a pronounced temperature gradient exists in the plating cells, making reported temperatures highly position dependent.

3. Fiber Synthesis Velocity Control

The velocity at which the fibers are synthesized, on the range of 0 - 12.7×10^{-3} mps (0 - 2.5 fpm), is controlled in incremental steps by inserting synchronous motors of various speeds into the take-up mechanism. This results in considerable accuracy in maintaining a fixed velocity. For somewhat less accurate synthesis velocities, a D. C. servo motor is employed. A simple traverse mechanism, utilizing a double helix cam, is coupled to the take-up spool drive to automatically lay up the fiber across the spool.

D. MONOFILAMENT EVALUATION

Fibers synthesized during this program are examined for diameter and

surface smoothness. General toughness in handling and tensile tests further classify the fibers. Synthesis parameters may then be altered based upon an engineering judgment of the quality of the fiber and a new experiment performed. When considered pertinent, fiber samples are examined for modulus, density, and elemental composition.

1. Diameter and Surface Smoothness

Fiber diameter and surface smoothness are assessed by optical microscopy at 400 \times . A Unitron inverted metallurgical microscope having a filar micrometer eye-piece is used for this purpose. For diameter measurement, the fiber is backlighted with an adjustable intensity lamp to produce a sharp profile. The filar micrometer is then adjusted to obtain three diameter readings and the results averaged. Surface smoothness is determined visually by observing the regularity of reflected light profile and by slowly moving the fiber in the field of view to detect nodules. Fibers having an excessive number of nodules are always weak. Higher strength fibers appear to have some latitude in surface texture ranging from glassy smooth to slightly irregular.

2. Density

Fiber density is determined by flotation. Two liquids of different densities, one having a density less than the fiber and one having a density greater than the fiber, are mixed in such proportion that a nominal 6 mm (1/4 inch) length of fiber will remain suspended therein after settling for 10 to 15 minutes. A precision hydrometer is then used to determine the liquid and, hence, fiber density. Liquid mixtures, as xylene and 2, 3-dibromopropanol are suitable for this purpose.

3. Preliminary Bend Testing

Experienced personnel can roughly classify fibers by simple manipulations, including bending the fibers over a finger tip, looping the fiber and

observing the loop radius at fracture, etc. Such elementary tests quickly assess general fiber quality and serve to screen fibers which are sufficiently strong and defect free so as to merit more rigorous testing methods.

4. Ultimate Tensile Strength

Material which appears promising in preliminary bend tests is tensile tested. Tensile tests are conducted by conventional load cell methods using a ten pound load cell coupled to a Statham transducer. Loading rate is 0.127 cm/min (0.05 in/min) on a 0.64 cm (0.25 in) gauge length. The testing is performed in a horizontal mode. The sample is affixed with epoxy to aligned 0.089 cm (0.035 in) diameter tungsten rods. Longer gauge lengths of 2.54 cm (1 in) were also studied.

5. Modulus

Modulus is determined by the dynamic "vibrating reed" technique. A length of the sample fiber (2.54 cm \pm 0.03 cm or 1.0 inch \pm 0.01 inch) is affixed at one end to a magnetic transducer driven by the amplified sine wave from an audio-frequency generator. The generator is calibrated by the Lissajous Figure method. The frequency is recorded at which maximum vibration occurs for the free end of the fiber. The fiber is glued to the transducer with household type cement. Diameter is measured by microscopy and density by flotation.

The modulus is determined from the relationship (Reference 22):

$$M = \frac{f^2 l^4 \rho}{7.58 d^2}$$

where l = length (in.)

M = elastic modulus (psi)

f = resonant frequency (Hertz)

ρ = density (lb/in³)

d = fiber diameter (inches)

Using this method, boron filament was found to have a modulus of 40 MN/cm² (58 \times 10⁶ psi), which is in agreement with accepted range of values.

6. Chemical Analysis

A limited number of filament samples were analyzed for boron content by Galbraith Laboratories, Knoxville, Tennessee. Galbraith Laboratories did attempt to analyze for carbon and hydrogen, but the analytical procedure did not give satisfactory results. The only elements present in the plating gas atmosphere are carbon, boron, hydrogen, and argon. Since only carbon and boron produce refractory solids, an analysis for boron content should yield carbon content by difference.

III. RESULTS & DISCUSSION

A. GENERAL

Previous contracts showed that large diameter carbon-based monofilaments can be made but that problems existed which prevented the successful attainment of a viable production process. These problems include scatter in strength, limitations in diameter and modulus, and substrate surface defects. Scatter in strength was found to be unpredictable and was believed to arise in part from random surface defects on the substrate which induced defective growth in the filament deposit. Random processing variations were also suspected and considerable effort was expended to assure maximum process control. However, some undefined overriding variable appeared to exist which prevented reduction in the scatter in filament strength. These previous contracts also showed that filament modulus was related to boron content, but the relationship was not quantified.

This program was organized on the basis of the filament property variables of strength, modulus, and diameter. Each filament property was studied to determine the critical variable or variables which have the prime effect on that fiber property and which have promoted successful and economical fiber production.

The findings of this program definitely establish that wide scatter in the carbon substrate strength is responsible for the heretofore inexplicable scatter in the monofilament strength. These findings also show that defective

substrate surface conditions which can induce low strength nodular growth in the monofilament layers are best controlled by processing improvements during the synthesis of the substrate. Modulus was found to be dependent on boron content (Figure 5). The results further show that filament modulus can be increased to above 27.6 MN/cm^2 ($40 \times 10^6 \text{ psi}$) but only by a considerable increase in boron content to 60 wt. % or more. This trend applies to alloy fibers having 30 to 35 wt. % boron or higher. Filament diameter depended, as might be predicted, upon the dwell time in the synthesis apparatus.

A filament was prepared using these findings which had the combined properties of a mean U.T.S. of $398,000 \text{ N/cm}^2$ ($568,000 \text{ psi}$), a modulus of 18.9 MN/cm^2 ($27 \times 10^6 \text{ psi}$), and a diameter of 145μ (5.71 mils). Highest measured strength for this fiber was $451,000 \text{ N/cm}^2$ ($645,000 \text{ psi}$).

B. EFFECT OF PROCESS VARIABLES ON TENSILE STRENGTH

1. Substrate

Substrate anomalies appear to be prime factors contributing to variations in the carbon base monofilament strength. This is documented by the results displayed in Table VIII. Table VIII shows the singular effect of changing substrate lots on ultimate tensile strength while holding all processing variables approximately constant. That undefined substrate variations have occurred between lots 1102 and 1132 is clear. It is also clear that these variations can have a deleterious effect upon filament processing.

It would seem that adjustment in process conditions can partially offset the deleterious effects of substrate lot change. Generally, it may take several weeks of fine alteration to affect this adjustment. For example, production of acceptable smaller diameter monofilament (ca. 90μ or 3.5 mil diameter) could be accomplished utilizing substrate lot 1132, but only after two weeks of fine changes in the synthesis parameters used when producing filament on the lot 1102 substrate. On the other hand, a substrate variation within a given lot could also cause this effect as it was being consumed during this adjustment period.

There does not appear to be radical change in electrical properties of

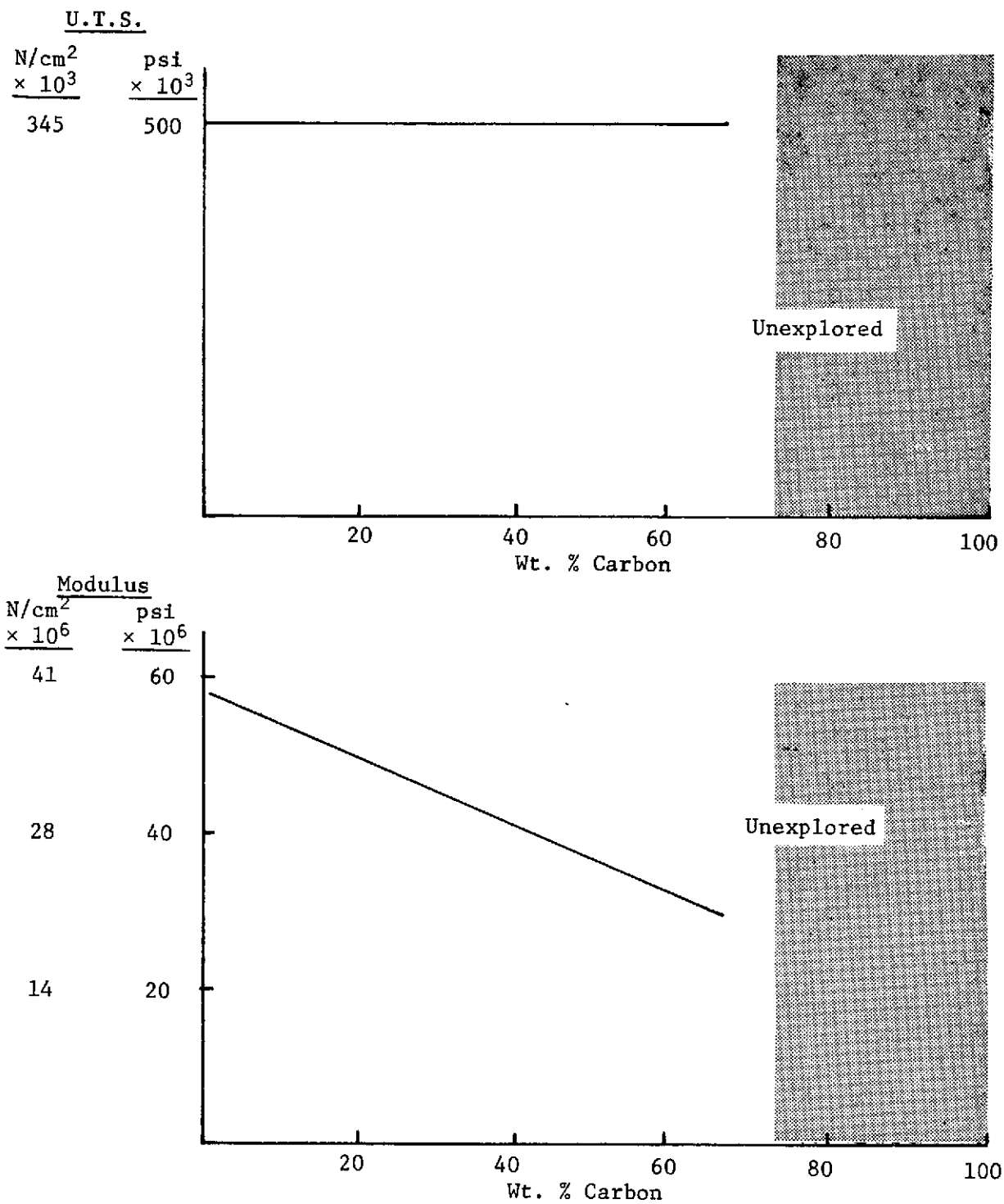


Figure 5 - Effect of Boron Content on Fiber Properties

the substrate from lot to lot as shown in Table IX. This data was obtained by monitoring the voltage - current parameters of the hydrogen cleaning cell at ca. 1400°C using a hydrogen flow of nominal .14 lpm and a hot zone length of 355 cm (14 in). While some change is noted in electrical resistivity and filament diameter, it does not appear to be the major overriding variable.

Microscopic observation of the substrate surface indicates that the Great Lakes Carbon Corporation has gained considerable experience in surface improvements, thus greatly reducing, though not totally eliminating, substrate surface defects as a possible variable. Rather, some other substrate problem seems to be the cause of carbon based CVD monofilament strength variation.

One suspect substrate problem is variation in the mechanical properties. Accordingly, we selected substrate samples from various lots that had been found to produce both acceptable (average $>206,000 \text{ N/cm}^2$ or $>300,000 \text{ psi U.T.S.}$) and unacceptable (average $<206,000 \text{ N/cm}^2$ or $<300,000 \text{ psi U.T.S.}$) CVD filament. Strain-to-failure, ultimate tensile strength, breaking load, and modulus were measured for these various lots. The tensile machine was fitted with a .91 kg (two pound) load cell and sample strain rate set at .13 cm/min (.05 in/min). A 2.54 cm (1 in) gauge length was used with sampling intervals of 231 to 354 cm (9 to 10 in) along the substrate. A 254 (10 in) gauge length with sampling at 457 to 540 cm (18 to 20 in) was also used for one lot (lot 1102). The longer gauge lengths were used to check for possible grip slippage effects on measured modulus. Lots 1070, 1084, 1102, 1132, 1133, 1144, and 1145 were tested, and the results are shown in Tables X and XI. Of these lots, nos. 1084, 1102, 1132, and 1144 produced acceptable CVD filament as defined above. There appears to be a correlation between strain-to-failure and acceptability. This strongly suggests that some strength limiting variable on or within the substrate is the responsible factor contributing to variations in the carbon based CVD monofilament strength. Studies at GLC indicate that surface defects, as nodules, do not seriously alter the carbon substrate strength or strain-to-failure. Thus, some internal and process related variable is evidently responsible. One possibility is that this variable may arise during the proprietary stabilization phase associated with the carbon substrate manufacture. That variations may also

occur within a given lot was also briefly documented by taking sufficient samples from the "poor" lot no. 1145 for a total sampling length of nominal 4.58 meters (14 feet). Significant strength variations were observed, as shown in Figure 6.

a. Effect of Etching Substrate

A series of experiments was performed to etch the Great Lakes carbon substrate. The main purpose was to etch away surface imperfections and irregularities that could develop into cracks and nodules on the finished fiber. A secondary anticipated effect of etching is to increase the strength of the finished fiber because of the volume % decrease of the relatively weak substrate ($<69,000 \text{ N/cm}^2$ or $<100,000 \text{ psi}$).

The experimental procedure used for etching is as follows: a short section of the moving substrate is electrically heated while a jet of the atmosphere being studied is impinged upon the hot zone. The different atmospheres tested for this process were:

1. Oxygen - argon
2. Argon
3. Hydrogen
4. Hydrogen - water vapor

Oxygen - Argon - An atmosphere of nominally 20% oxygen in argon was used, but this proved to be so reactive that it was impossible to prevent the substrate from burning through. To circumvent this, it was necessary to initiate the heating in a pure argon atmosphere and then to gradually introduce the oxygen until the experimental concentration was achieved. Even using this precaution, the entire system was too unstable and sensitive to provide a suitable etch environment.

Argon - An atmosphere of pure argon was investigated next. In a series of five experiments using the temperature range of $1200 - 1600^\circ\text{C}$, we were unable to obtain any reduction in diameter. Therefore, pure argon was abandoned as a potential etch gas.

Hydrogen - The use of pure dry hydrogen provided a very stable system, but, unlike argon, we were able to obtain a definite etching of the substrate.

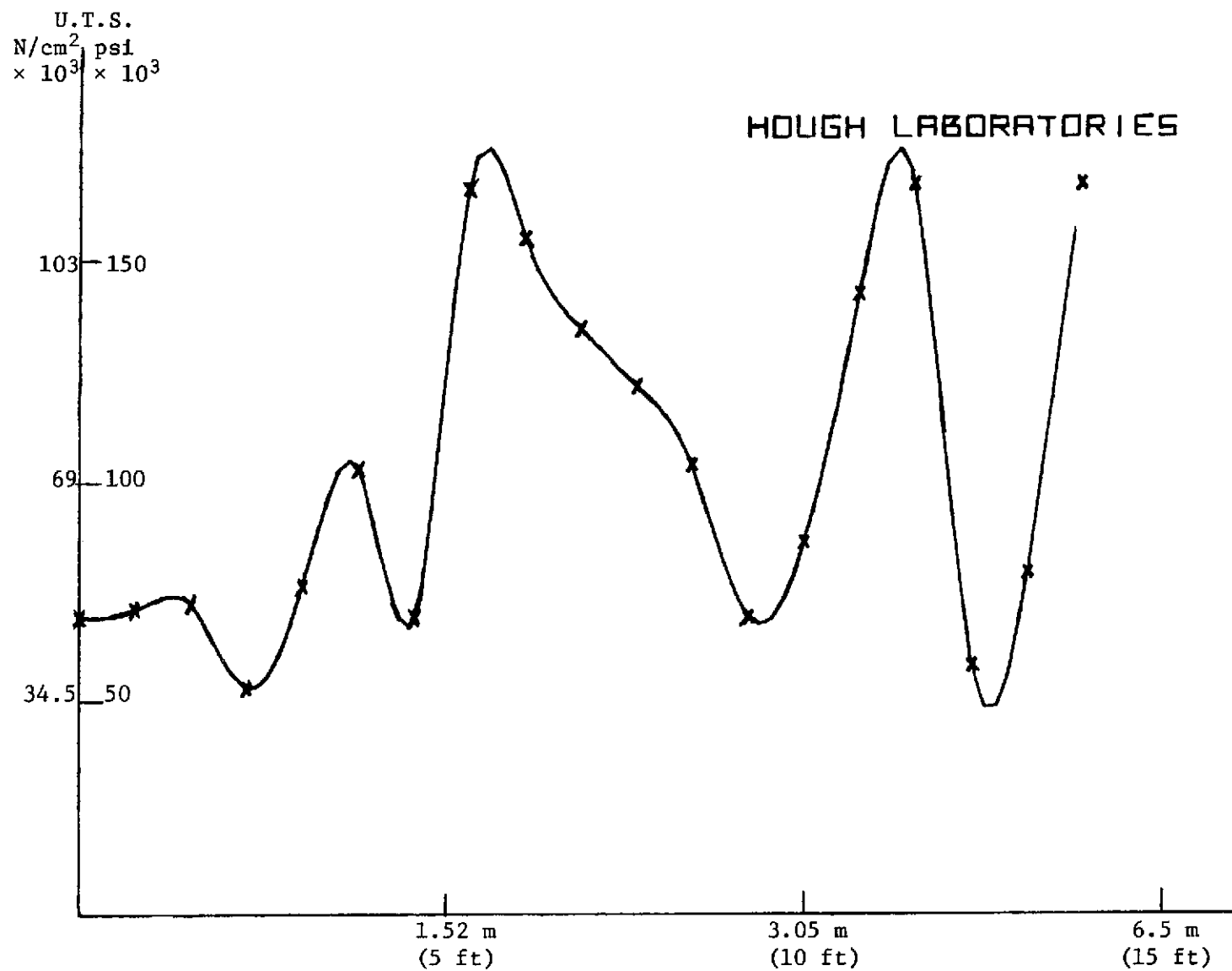


Figure 6 - Substrate Strength Variations with Length
(Lot 1145, Spool 1)

Several experiments were performed, and all showed significant reductions in the substrate diameter. The most significant results were obtained at 1030°C and were as follows:

Initial diameter	33.3 μ (1.31 mils)
Final diameter	30.7 μ (1.21 mils)
% Cross-sectional area decrease	14.7%

Hydrogen etching differs from hydrogen cleaning described later only in that insufficient time is allowed for the carbon surface to be significantly removed.

Hydrogen - Water Vapor - The final atmosphere studied was hydrogen bubbled through distilled water at room temperature. This system was very stable, and the results were even more encouraging than with dry hydrogen. Therefore, an additional series of experiments was performed on this system.

In the first series, the velocity was held constant at .008 m/sec (1.58 fpm) while different temperatures were studied to determine the effects of temperature. Results are displayed in Table XII.

In the second series of experiments, the temperature was maintained at an arbitrary value (1300°C) while the filament velocity was varied. The purpose of this series was to determine if the etching followed a standard kinetic rate equation. Results are displayed in Table XIII. For this data displayed, the standard first order rate equation was found to apply. This corresponds to related work by Hedden (Reference 23). Using the exponential form of the rate equation $d_2^2 = d_1^2 e^{-kt}$, final diameters can be calculated for the experimental velocities. Table XIV illustrates the correspondence between calculated and experimental final diameters.

Plating experiments using the etched fiber did not produce any improvement in fiber strength or modulus. The surfaces of the plated fibers produced without an etched substrate appeared as smooth or smoother than filaments deposited on the etched substrate. We conclude that etching is not warranted and may actually increase filament synthesis problems by uncovering occlusions beneath the "as received" substrate surface.

Several experiments were performed using the H₂ - H₂O etch process described above. The etched substrate was used to produce carbon-base monofilament. Table XV illustrates the results of these experiments. Using the

same plating atmospheres as the others, experiments 142 and 145 were run without etching the substrate. As can be seen in the results columns, the etched fiber exhibits no marked advantage over the unetched material.

b. Effect of Precoating Substrate

In an attempt to eliminate some of the scatter of our data, we ran a series of tests to determine the effect of a thin intermediate coating on the carbon substrate. One of the first systems tested used trichlorosilane to deposit elemental silicon. Although the fiber was apparently coated, the resulting coating was too rough and brittle. Several organo-silanes were tested, in an attempt to plate SiC. These all resulted in the same type of rough, brittle coat. Next, an ethylene plating atmosphere was used to try plating carbon onto the H₂ cleaned substrate. In this system, a smooth, adherent coating was plated on the 33 μ (1.3 mil) substrate to increase the diameter to 38 μ (1.5 mils). Using this precoat, tests were again conducted with the boron trichloride - methane system. The results are displayed in Table XVI.

The carbon precoat was then used on the triethyl borane - ethylene system. Table XVII displays the results of this series of experiments. It should be noted that the carbon coated substrate represents a process modification which we believe to be a correct step, and that this modification requires process modifications to accommodate the change.

It appears that a precoating of carbon may help to "standardize" the carbon substrate surface, and may therefore hold promise in solving any substrate surface defect problem. The precoating of carbon also helps to reduce thermal gradients in the main plating cells. This is an important factor which may enhance long term production run capacity by reducing secondary deposition on the reactor walls.

Plating gas conditions which yielded reasonably good quality carbon alloy filament when utilizing carbon substrates were applied in experiments utilizing both 12.7 μ (0.5 mil) and 25.4 μ (1.0 mil) diameter tungsten wire substrates. The carbon-to-boron ratio for triethyl borane and ethylene was held at 66:1 with a 20% reactive gas concentration. Plating temperature was

varied over the range of ca. 1000° to 1250°C. Results are shown in Table XVIII. All the fibers using tungsten substrates were weak. Best results occurred at the lower temperatures, which also produced smoother fiber surfaces. No fibers were obtained having tensile strengths above 138,000 N/cm² (200,000 psi) using the tungsten substrate. Fiber strengths for the carbon substrate control fibers were in excess of 206,000 N/cm² (300,000 psi) for fibers of comparable final diameters.

It is known that during the deposition of pyrolytic carbon upon tungsten filament, a contiguous interfacial zone is generally not achieved because of extensive interdiffusion of both tungsten and carbon (Reference 24). Evidently, a sufficient concentration of carbon is present in the deposits to preclude successful high quality filament preparation. Filaments containing higher concentrations of boron are predicted to have more contiguous interfacial zones. However, no guarantee can be made that such fibers will be of high quality. For example, the average tensile strength for boron carbide filament deposited upon tungsten substrate from mixtures of ethane and boron trichloride in hydrogen is reported as only 181,000 N/cm² (263,000 psi) (Reference 25). We have, therefore, concluded that tungsten as a substrate is not at present an attractive approach.

2. Process Parameters

a. Effects of Temperature

The effects of plating temperatures for the organoborane - ethylene gas system were studied for multiple plating cells. Simultaneous changes in the temperatures of four plating cells in a five stage line were studied utilizing untreated substrate. The first stage is operated as a hydrogen cleaning cell to remove volatile surface contaminants from the substrate. Results, shown in Table XIX, suggest that 1100°C for the second stage and 1000°C for subsequent stages is preferred.

To further substantiate this conclusion, experiments were performed in which the temperature of the second stage was varied while holding subsequent stage temperatures at 1000°C. These experiments were performed at a nominal

fiber velocity of 0.0123 m/sec (2.43 fpm) in a plating gas atmosphere having a boron-to-carbon ratio of ca. 1:66 and with a total reactive gas concentration of nominal 10%. Results of this series of experiments are displayed in Table XX.

The results strongly suggest that preferred plating temperatures for the triethylborane - ethylene gas system are ca. 1100°C for the first plating cell and 1000°C for subsequent plating cells.

b. Effect of Gas Composition

Reactive Gas Concentration - Table XXI presents data on the effects of varying the total reactive gas concentration while maintaining relatively constant total gas flow rate. The boron-to-carbon ratio is maintained at nominal 1:66. In these experiments, the substrate was not etched but merely precleaned in hydrogen in stage 1 at a temperature of 1400°C. Both two and three plating cells were utilized as shown in the table. There appears to be some advantage in utilizing nominal 20% total reactive gas concentration. The argon functions as a diluent to moderate the reaction, and the hydrogen serves to suppress excessive pyrolytic soot formation.

Carbon-to-Boron Ratio in Triethylborane - Ethylene System - The carbon-to-boron ratio was varied on the range of approximately 60:1 to 70:1. In all experiments, stage two was at 1100°C, and subsequent stages were at 1000°C. Filament synthesis was performed utilizing both two and three plating cell lines. Results of these experiments are displayed in Table XXII. All the experiments were performed at a filament synthesis velocity of .0038 m/sec (0.75 fpm) with the exception of experiment 183, which had a velocity of .0051 m/sec (1.0 fpm).

Additional experiments were performed utilizing the organoborane - ethylene plating atmosphere in which the carbon-to-boron ratio was examined over the range of 40:1 to 100:1. In these experiments, synthesis velocity was increased to .0051 m/sec (1.0 fpm) while using both two and three plating cell lines. These experimental results are shown in Table XXIII. We note that the overall modulus level appears to have decreased over that obtained at the lower synthesis velocity. Also, we note that the general modulus level

appears somewhat higher for two as opposed to three plating cell line operation.

We conclude that a C:B ratio of 66:1 is suitable for the organoborane - ethylene gas system although a distinct optimum point does not seem to occur.

Boron Hydride - Ethylene Gas System - The effect of diborane - ethylene gas compositions on fiber tensile strength and modulus was studied. Utilizing a three cell synthesis line and a filament velocity of .0051 m/sec (1.0 fpm), we varied the carbon-to-boron ratio in the plating gas on the range of 40:1 to 100:1. Plating temperatures were 1400°C in the cleaning cell, 1100°C for Stage 1, and 1000°C for Stage 2. Best results appear at a 54:1 ratio for the diborane - ethylene gas system (see Table XXIV). Also of interest is the somewhat higher modulus values obtained compared to organoborane - ethylene. Additional work was also performed to relate C:B ratio, number of plating cells, and filament velocity to both strength and modulus. Results are shown in Table XXV. These results tend to confirm the 54:1 ratio. Also, there appears to be some advantage using two cells as opposed to three and for using a filament velocity of .0038 m/sec (0.75 fpm). There is also a trend toward higher modulus compared to the organoborane - ethylene system.

In the above reported work, we find that all the filament property levels are interrelated. Parameters which are altered to attempt to change tensile strength also affect both modulus and diameter. Thus, in all the above experiments, we note a considerable increase in filament diameter over previous work.

Boron Trichloride - Hydrocarbon Plating Gas - When studies of this gas system were first undertaken, the initial results appeared very encouraging. However, as work progressed, it became apparent that these gas mixtures might not be usable in a viable full scale production system. The preliminary work showed excellent properties, U.T.S. over 276,000 N/cm² (400,000 psi) and modulus of nominally 27.6 MN/cm² (40×10^6 psi). In actual trial production runs, on the order of 15.3 to 30.5 meters (50 to 100 feet) or more, the gas system exhibited very different characteristics. Table XXVI illustrates these poor results. Although the modulus is high, tensile strength values are not consistently good. In addition, the density of the material which is over 2.2 g/cc reflects very high boron content and might be considered in some respects more a modified boron filament than a modified carbon fiber.

There appears to exist an inherent instability problem associated with the synthesis of filament utilizing a plating gas system based on boron trichloride and hydrocarbon. While this instability does not preclude the attainment of high tensile strength values, it does appear to seriously limit the probability of consistently synthesizing high quality filament. This instability is in all probability directly related to the chemical intermediates that can form upon pyrolysis of a gas system which simultaneously contains both boron halide and hydrocarbon gases. While other plating gas systems containing boron hydride or organoboranes and hydrocarbon can form intermediates, such intermediates are not believed to behave in as deleterious a manner as those produced in the boron trichloride based plating gas system.

The cited instability of the boron trichloride - hydrocarbon plating gas system is probably related to the formation of intermediates. Thus, Gipstein and workers (Reference 26) show that the vapor-phase reaction between acetylene and boron trichloride forms β -chlorovinylboranes. The compound 2-chlorovinylldichloroborane quickly forms. In excess acetylene and in the presence of mercuric chloride and activated carbon, bis-(2-chlorovinyl) chloroborane and some tris-(2-chlorovinyl)borane may form. Traces of mercury halides and active carbon will invariably be present in a chemical vapor deposition apparatus which uses a boron trichloride - hydrocarbon atmosphere. The mercury halide comes from mercury vapor given off by the mercury electrode contacts. Even if electrodeless heating methods are utilized to eliminate the mercury, 2-chlorovinylldichloroborane will still form. The acetylene, if not initially present, will be formed by partial pyrolysis of the hydrocarbon whether the hydrocarbon is methane, ethane, ethylene, etc. (Reference 27). That McCandless and others (Reference 25) could not produce high strength boron - carbon fibers utilizing boron trichloride - acetylene lends credence to the possibility that intermediates, as 2-chlorovinylldichloroborane, may mitigate against a successful filament production process development. The action of these and other boron trichloride derived intermediates may include undesirable formation of dielectric polymeric residues near electrode contacts which can induce arcing and, hence, random weakened filament areas. We have indeed observed a pronounced trend toward arcing for boron halide - hydrocarbon gas systems compared to the organoborane - hydrocarbon systems.

Another action of the intermediates might be to induce random and uncontrollable microcrystalline low strength areas. Similar strength limiting mechanisms are known to exist for boron filament synthesis when carbonyl chloride or hydrogen chloride are present in the initial plating gases (Reference 28). Gatti, Cree and Feingold (Reference 29) also failed to prepare consistently high quality boron - carbon filament when working with the gas system: hydrogen, methane, boron trichloride.

For these reasons, we rejected the boron trichloride based plating gas system. It does not appear to be an attractive approach for achieving a viable carbon-base filament production process.

Selected filaments are characterized for boron content to determine the effect of this variable on tensile strength and modulus. Table XXVII shows results. As a general rule, modulus increases as boron content increases.

c. Effect of Drawing Speed

Changes in drawing speed do not effectively change U.T.S. This effect may be observed from the data displayed in Table XXV.

As is evident in the foregoing discussions, there are various combinations of parameters that result in reasonably high physical properties for the final fiber. It should be stated again that the main factor affecting the fiber strength is actually the strength of the substrate used. If the substrate is of reasonably consistent strength - on the order of $89,600 \text{ N/cm}^2$ (130,000 psi) - we were able to obtain fairly high monofilament strength using most of the gas systems utilized.

G. EFFECT OF PROCESS PARAMETERS ON MODULUS OF ELASTICITY

1. Substrate

Based upon a rule of mixtures consideration, the low modulus of the carbon substrate - 3.4 MN/cm^2 (4.9×10^6 psi) - might have a modulus diluting effect for the final monofilament. However, Table XV illustrates that we were unable to produce any marked increase in the modulus through utilization

of an etch process which reduced the size of the substrate cross-section. No conditions were found in which the substrate produced an appreciable effect on fiber modulus.

2. Process Parameters

a. Effects of Temperature

During our work, we have found that the higher the plating temperature, the higher the carbon content in the fiber when using organoborane - ethylene. Conversely, the cooler the plating process, the higher the boron content. When one examines Figure 8, it is seen that the higher the weight per cent boron in the fiber, the higher the modulus, on the range of 30 to 100%. The range below 30% boron is an unexplored region. Table XXVII also illustrates this effect of boron content on fiber modulus. Unfortunately, there is only a narrow temperature band that can be used and still produce fibers whose other properties, strength and diameter, are also acceptable. Table XX illustrates the effect on modulus of first plating cell temperature change. Higher temperatures decrease modulus. Also illustrated is the effect that these temperatures have on U.T.S. Any deviation either up or down in temperature causes a drop in strength.

b. Effects of gas composition

As has been illustrated, the modulus is directly related to the amount of boron in the fiber. Table XXVII also illustrates for both the boron hydride and the organoborane system that, at a given temperature, the boron in the fiber is dependent upon the C:B ratio in the plating atmosphere. Table XXIV shows that for the diborane system, the modulus decreases with the increased C:B ratio in the plating gas. Table XXVI illustrates that this same relationship holds true for the boron trichloride system.

The modulus of carbon - boron fiber can be increased by increasing the amount of boron in the fiber. Boron content increases with both temperature decrease and boron-to-carbon ratio increase. Unfortunately, as the boron

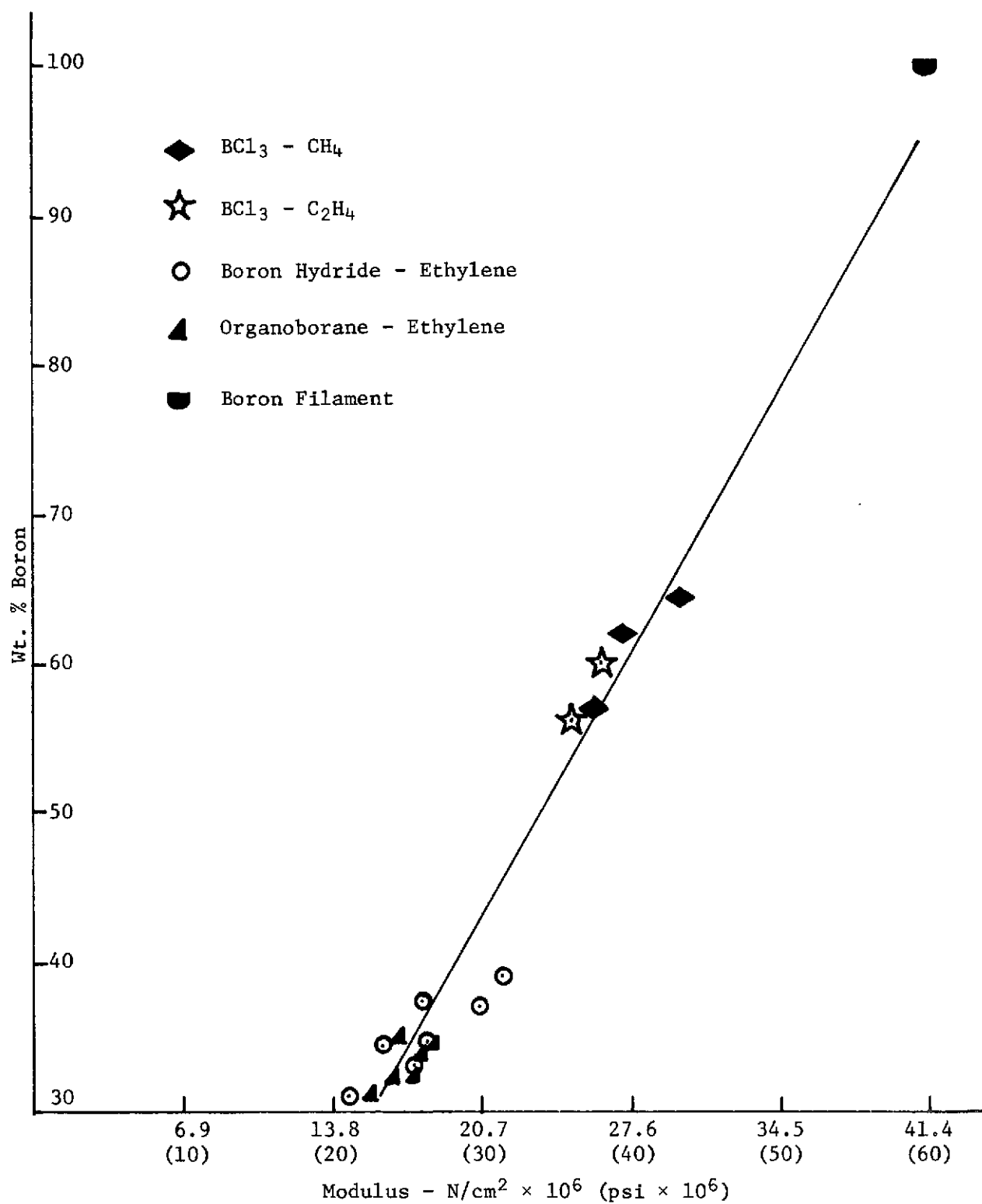


Figure 7 - Monofilament Boron Content vs. Modulus

content increases, the density of the fiber also increases. In sample fibers with modulus approaching 27.6 MN/cm^2 ($40 \times 10^6 \text{ psi}$), the densities were 2.2 g/cc or higher. To increase the modulus much over 20.7 MN/cm^2 ($30 \times 10^6 \text{ psi}$) means to sacrifice the attractive low density of carbon fiber.

c. Effects of Other Variables

Other parameters and variables appear to have little effect on the fiber modulus. Filament velocity experiments are illustrated in Table XXV for various C:B ratios. There is apparently no velocity that produces higher moduli for all C:B ratios studied.

D. EFFECTS OF PROCESS PARAMETERS ON DIAMETER

The deposition of carbon - boron alloy material occurs when boron and carbon compounds are thermally decomposed in the reaction chamber. Any parameter change that increases either the dwell time or the reaction rate will obviously increase the amount of material deposited and, hence, increase monofilament diameter. Dwell time is easily increased by increasing the chamber lengths, increasing the total number of cells, or decreasing the synthesis speed. Previous work (Reference 20) has shown that diameter approximates a function of the square root of dwell time. The reaction rate can be increased by increasing the temperature, the concentration of the reactants, or by using a reactant gas system that may have a higher decomposition rate than those we have studied.

1. Effect of Drawing Speed

By holding other parameters constant and decreasing filament speed, larger diameter fiber can be produced. In Table XXV, the diameters produced by various drawing speeds can be compared for a number of carbon-to-boron ratios. In one unrecorded run at .0041 m/sec (0.8 fpm), we have produced a 193μ (7.8 mil) fiber. The obvious disadvantage to this is the reduction in footage per unit time. This is compensated for by the increased weight per

unit length.

2. Effect of Plating Temperature

At a constant speed, increasing the temperature of the reaction increases the rate at which the alloy material is deposited. Unfortunately, temperature changes also affect the quality of the plated material. Table XX illustrates corresponding diameter increases with temperature increases. Also shown are the reduced modulus and strength resulting from the temperature increase.

3. Effect of Reactant Gas Concentration

As shown in Table XXI, concentration of the reactive gases has a minimal effect on the physical properties of the fiber.

4. Effect of Other Variables

Lengthening the plating cell lengths also increases the final diameter. But, as the length of a cell increases, the power required to heat the fiber to the proper temperature also increases. This power consideration makes increased lengths impracticable using existing equipment.

Another approach is to increase the number of plating cells. This allows for shorter cell lengths and lower voltages but increases the number of power systems and gas distribution systems needed. The amount of gas needed is also increased. The effect of changing cell number on final diameter is displayed in Table XXIII.

E. SUMMARY OF EFFECT OF PROCESS VARIABLES ON PROPERTIES

The effects of process variables on ultimate tensile strength, modulus and diameter have been discussed and the results given. Each of these monofilament properties was studied to determine the critical variable or variables which have prevented successful and economical fiber production.

With the exception of filament drawing speed, the best synthesis

parameters for preparing 75 to 100 μ (3 to 4 mil) diameter monofilament also apply to nominal 150 μ (6 mil) diameter monofilament. The preferred plating gas was found to be a 20 volume per cent mixture of triethylborane - ethylene (Table III) in hydrogen - argon. We used a carbon-to-boron ratio of 66:1, but the ratio was not particularly critical. Plating temperatures are critical. Plating temperatures of 1100°C for the first plating cell and 1000°C for any subsequent cell are necessary (Table XIX). The gas flow distribution was found to be important for multiple cell systems. Thus, for two plating stage operation, the first plating stage requires nominal 45%, the second stage 55%, of the total gas flow (Table VII). For three plating stages, the ratios are 28.6:35.7:35.7.

The critical overriding variables controlling monofilament strength, modulus and diameter were found to be the substrate, the boron content of the fiber, and the drawing speed respectively.

The findings of this program definitely establish that scatter in the strength of the substrate is the critical variable regulating monofilament strength. In addition, the strength level of various substrate lots varies considerably (Table X). Substrate having a mean tensile strength below 90,000 N/cm² (130,000 psi) did not consistently produce monofilament having a tensile strength above 206,000 N/cm² (300,000 psi). The findings of this program also show that defective substrate surface conditions which can induce low strength nodular growth in the monofilament layers are best controlled by processing improvements during the synthesis of the substrate rather than by etching.

The critical variable regulating modulus is filament boron content for alloy fiber having 30 to 35 weight per cent boron or higher. Thus, the filament modulus can be increased to above 27.6 MN/cm² (40×10^6 psi), but only by a considerable increase in boron content to 60 weight per cent or higher. The critical variable regulating diameter was found to be filament drawing speed. At drawing velocities of .008 m/sec (1.5 fpm), filaments of nominal 88.9 μ (3.5 mil) were obtained using three plating cells. Comparable results were obtained at .013 m/sec (2.5 fpm) using four plating cells. By comparison, at .004 m/sec (0.75 fpm) using two plating cells, fibers of 140 to 152 μ (5.5 to 6.0 mil) diameter were prepared.

Table XXVIII displays the best combination of ultimate tensile strength, modulus, and diameter obtained during this effort and compared to the two previous efforts. High modulus values can be obtained by synthesizing the monofilament as a modified boron fiber.

F. COST REDUCTION, QUALITY CONTROL AND PRODUCTION CAPABILITY

1. Cost Reduction.

Table XXIX displays projected cost information on large diameter (142 μ or 5.6 mil) carbon alloy monofilament. The projected cost of \$97.74/lb. is based entirely upon current state-of-the-art and does not include potential cost savings through recycling gases.

What is more significant from the standpoint of further cost reduction is the possibility of reducing or eliminating the boron content. By doing this, we can eliminate the triethylborane and the argon. We can also adjust downward the hydrogen requirement. Thus, it becomes possible to predict combined materials costs and utilities per pound of large diameter carbon fiber at ca. \$25.00 or less. It then becomes feasible to consider costs below \$50.00 per pound of fiber. Thus, lower potential costs for large diameter carbon-base monofilaments are less than those of boron. In addition, hydrocarbon starting materials, as ethylene at \$.04 per pound are available multisource, whereas boron trichloride is available from the sole source, Kerr-McGee; it is in limited supply and costs \$1.80 per pound (Reference 30).

2. Quality Control

Through the process of elimination, the problems affecting quality control have been isolated as strength and strength scatter in the carbon substrate. This factor was determined only late in the contract period. However, through optimization of the processing parameters, good quality control was obtained, contingent upon the overriding variable of substrate affects. Generally, quality control appears better for the larger diameter monofilament. Table XXX shows production information on 1524 meters (5000 feet) of

75 to 100 μ (3.0 to 4.0 mil) diameter monofilament. Table XXXI shows similar information for 1066 meters (3500 feet) of nominal 142 μ (5.6 mil) diameter monofilament.

3. Production Capability

Currently, production line modules have the capability of producing a maximum of 305 meters (1000 feet) of 142 μ (5.6 mil) fiber or 914 meters (3000 feet) of nominal 86.4 μ (3.4 mil) fiber per day.

IV. CONCLUSIONS

The primary conclusions reached during this program are:

1. The CVD method for producing large diameter carbon-based monofilament was further developed by studying the independent effects of process variables on filament strength, filament modulus, and filament diameter. The work was intended to isolate those overriding variables which control fiber properties and which had not been defined in previous efforts. One of the more important variables controlling monofilament strength was found to be the wide variation in the strength of the Great Lakes carbon substrate. Not only does the strength variation occur from lot to lot, but also within a given lot over short spans on the order of 3 meters (10 feet) or less. This strength variation for the substrate is believed to be related to internal flaws which arise during the proprietary stabilization step in their manufacture.

2. Filament modulus was found to be nearly linearly dependent upon boron content for the range studied, namely 30 to 35 weight per cent boron and above.

3. Monofilament diameter depends upon dwell time in the synthesis apparatus. Previous work (Reference 20) has shown that diameter approximates a function of the square root of dwell time.

4. Using the findings of this effort, a monofilament was prepared having the combined properties of a mean U.T.S. of 398,000 N/cm² (568,000 psi) on 2.54 cm (1 in) gauge length, a modulus of 18.9 MN/cm² (27×10^6 psi) and a diameter of 145 μ (5.71 mils). Highest measured strength for this fiber was

451,000 N/cm² (645,000 psi) on 2.54 cm (1 in) gauge length. By comparison, the previous experimental high was 381,000 N/cm² (552,000 psi) on .68 cm (1/4 in) gauge length for a fiber of nominal 89 μ (3.5 mil) diameter. The largest diameter demonstrated during this effort was 193 μ (7.6 mil) compared to a previous high of 152 μ (6.0 mil).

5. The carbon portion of the carbon-boron alloy large diameter monofilament appears to have high strength independent of boron content for boron content of nominal 30 to 35 weight per cent and above. Modulus is, however, a direct function of boron content. No significant improvement was obtained for the monofilament modulus value because it was not considered advisable to further increase boron content.

R E F E R E N C E S

1. Schestzenberger, P. and Schestzenberger, L., "Sur quelques faits relatifs á l'histoire du carbone," Compt. Rend. 111, 774-778 (1890).
2. Constant, M. and Pelabon, H., "Sur une varie'ti' de carbone filamentlux," Compt. Rend. 137, 706 (1903).
3. R. Bacon, "Growth, Structure, and Properties of Graphite Whiskers," J. Appl. Phys. 31.2, 283-290 (Feb. 1960).
4. R. Bourdeau, private communication, August 1964.
5. F. E. Papalegis and R. G. Bourdeau, "Pyrolytic Reinforcing Agents for Ablative Erosion-Resistant Composites," ASD-TDR-63-403, W-PAFB, Ohio (May 1963).
6. F. E. Papalegis and R. G. Bourdeau, "Pyrolytic Reinforcements for Ablative Plastic Composites," ML-TDR-64-201, W-PAFB, Ohio (July 1964).
7. R. G. Bourdeau, private communication, August 1969.
8. R. Bacon, "High Strength - High Modulus Carbon Fibers," AFML-TR-66-334, Part I, W-PAFB, Ohio (Dec. 1966).
9. S. Otani, "On the Carbon Fiber from the Molten Pyrolysis Products," Carbon 3, Pergamon Press Ltd., Great Britain, 31-38 (1965).
10. W. R. Benn, private communication, June 1969.
11. E. R. Stover and R. R. Berning, "Effects of Strain-Annealing on Structure and Mechanical Properties of Pyrolytic Graphite," Paper No. 111, Fifth Biennial Conference on Carbon, The American Carbon Committee, University Park, Pennsylvania (June 1961).
12. R. L. Hough, "Continuous Pyrolytic Graphite Filaments," AFML-TR-64-336, W-PAFB, Ohio (Dec. 1964).
13. R. L. Hough, "Continuous Pyrolytic Graphite Composite Filaments," J.AIAA 3.2, 291-296 (Feb. 1965).
14. R. H. Clinard, Texaco Experiment Inc., private communication, October 1967.

15. R. H. Clinard, Texaco Experiment Inc., private communication, September 1969.
16. N. E. Quackenbush, "Large Diameter Graphite/Carbon Composite Filament Development," NASA CR-71769, Contract NAS3-13204 (July 1970).
17. W. G. Bradshaw, P. C. Pinoli, and A. E. Vidoz, "Development of Manufacturing Process for Large-Diameter Composite Monofilaments by Pyrolysis of Resin-Impregnated Carbon-Fiber Bundles," NASA CR-120973, Contract NAS3-15552 (Oct. 1972).
18. G. A. Cooper, D. G. Gladman, J. M. Sillwood, and G. D. Sims, "The Development of Composite Carbon Fibers of Large Diameter: Jumbo Fibers," NPL-IMS-18, National Physical Laboratory, Teddington, England (Nov. 1972).
19. R. L. Hough, "Development of Manufacturing Process for Large Diameter Carbon-Base Monofilament by Chemical Vapor Deposition," NASA CR-72770, Contract NAS3-12429 (Nov. 1970).
20. R. L. Hough and R. D. Richmond, "Improvement of Chemical Vapor Deposition Process for Production of Large Diameter Carbon Base Monofilaments," NASA CR-120902, Contract NAS3-14325 (Nov. 1971).
21. P. E. Elkins, G. M. Mallan and H. Shimizu, "Modified Silicon-Carbide Continuous Filaments," paper D-41, SAMPE, Vol. 10, November 1966.
22. J. A. Alexander, A. L. Cunningham, and K. C. Chuang, "Investigation to Produce Metal Matrix Composites with High-Modulus, Low-Density Continuous-Filament Reinforcements," AFML-TR-67-391, W-PAFB, Ohio, Contract AF33(615)-2862 (Feb. 1968).
23. K. Hedden, Proc. Fifth Conf. on Carbon, Vol. 1, 125, Pergamon Press, New York (1952).
24. R. L. Hough, unpublished work.
25. L. C. McCandless, et al, "High Modulus-to-Density Fiber Reinforcements for Structured Composites," AFML-TR-65-265, Part II, W-PAFB, Ohio (Sept. 1966).
26. E. Gipstein, P. R. Kippur, M. A. Higgins, and B. F. Clark, J. Org. Chem. 26, 2947 (1961).
27. Powell, et al, Vapor Deposition, John Wiley & Sons, Inc., New York, 352-356 (1966).

28. L. E. Line, Jr. and U. V. Henderson, Jr., Handbook of Fiber-glass & Advanced Plastics Composites, Geo. Lubin (Ed.), Van Nostrand Reinhold Co., New York, 226-227 (1969).
29. A. Gatti, R. Cree, E. Feingold, Genl. Elec. Co., Space Sciences Laboratory, Philadelphia, Pennsylvania, private communication, 1964.
30. L. Erikson, Kerr-McGee Chemical Corporation, private communication, April 11, 1973.

TABLE Ia
METHANE-BORON TRICHLORIDE PARAMETERS
(SI Units)

Exp. #	Flow Rates (ml/min)				B:C Ratio	Velocity mps $\times 10^{-3}$	Diameter μ	Avg. U.T.S. ¹ N/cm ² $\times 10^3$	Modulus N/cm ² $\times 10^6$
	Ar	BCl ₃	H ₂	CH ₄					
258	950	154	657	371	1:2.4	3.9	114 ²	226	27
259	950	154	657	371	1:2.4	3.9	113 ²	285	34
273 ³	1192	154	793	55.8	2.7:1	3.8	134	169	26
274 ³	1192	154	793	46.5	3.3:1	3.8	140	145	27
278	950	154	657	204	1:1.3	3.8	129	210	26
279	950	154	657	371	1:2.4	4.3	117	141	29
280	950	154	657	371	1:2.4	4.3	116	130	30

¹ All Gauge Lengths = 2.54 cm

² See Table V

³ C₂H₄ replaced CH₄

Cell lengths: H₂ - 33 cm; #1 - 94.6 cm; #2 - 116.8 cm

TABLE Ib
METHANE-BORON TRICHLORIDE PARAMETERS
(English Units)

Exp. #	Flow Rates (ml/min)				B:C Ratio	Velocity fpm	Diameter (mils)	Avg. U.T.S. ¹ psi × 10 ³	Modulus psi × 10 ⁶
	Ar	BCl ₃	H ₂	CH ₄					
258	950	154	657	371	1:2.4	.77	5.68 ²	324	39
259	950	154	657	371	1:2.4	.77	4.43 ²	408	48
273 ³	1192	154	793	55.8	2.7:1	.75	5.26	242	35.9
274 ³	1192	154	793	46.5	3.3:1	.75	5.5	208	38.2
278	950	154	657	204	1:1.3	.75	5.09	301	37.5
279	950	154	657	371	1:2.4	.85	4.62	202	42
280	950	154	657	371	1:2.4	.85	4.56	182	43.5

¹ All Gauge Lengths = 1"

² See Table V

³ C₂H₄ replaced CH₄

Cell lengths: H₂ - 13"; #1 - 37-1/4"; #2 - 46"

TABLE II-a
DIBORANE SYSTEM GAS PARAMETERS¹
(SI Units)

<u>Exp. #</u>	<u>Flow Rates (ml/min)</u>				<u>C:B Ratio</u>	<u>Velocity mps $\times 10^{-3}$</u>	<u>Diameter (μ)</u>	<u>Avg. U.T.S.² N/cm² $\times 10^3$</u>	<u>Modulus N/cm² $\times 10^6$</u>
	<u>Ar</u>	<u>B₂H₆</u>	<u>H₂</u>	<u>C₂H₄</u>					
208	1206	7.3	648	378	1:52	3.8	126	226	21
209	1268	7.3	544	378	1:52	3.8	131	225	20
211	854	3.7	657	378	1:102	4.0	102	126	18
214	972	5.2	498	378	1:73	3.8	116	152	19
220	1023	9.1	407	372	1:41	3.8	137	182	18
230	1153	6.8	634	372	1:55	3.8	127	242	21
234	1153	6.8	634	372	1:55	5.1	114	193	19
237	1153	6.8	634	372	1:55	7.6	98	147	18
245	1045	6.8	993	459	1:68	5.1	110	189	19
250 ³	1734	10.0	952	567	1:57	3.8	171	163	19

¹ See Table VI

² All gauge lengths = 2.54 cm

³ Three plating cells

Cell Length: H₂ 33 cm; #1 94.6 cm; #2 116.8 cm; #3 115.7 cm

TABLE II-b
DIBORANE SYSTEM GAS PARAMETERS¹
(English Units)

<u>Exp. #</u>	<u>Flow Rates (ml/min)</u>				<u>B:C</u> <u>Ratio</u>	<u>Velocity</u> <u>fpm</u>	<u>Diameter</u> <u>mils</u>	<u>Avg. U.T.S.</u> ² <u>psi × 10³</u>	<u>Modulus</u> <u>psi × 10⁶</u>
	<u>Ar</u>	<u>B₂H₆</u>	<u>H₂</u>	<u>C₂H₄</u>					
208	1206	7.3	648	378	1:52	.74	4.95	324	30.7
209	1268	7.3	544	378	1:52	.74	5.15	322	29
211	854	3.7	657	378	1:102	.78	4.02	180	25.6
214	972	5.2	498	378	1:73	.74	4.57	218	27.2
220	1023	9.1	407	372	1:41	.74	5.4	260	26.2
230	1153	6.8	634	372	1:55	.75	5.01	347	29.4
234	1153	6.8	643	372	1:55	1.0	4.49	277	27.6
237	1153	6.8	630	372	1:55	1.5	3.82	210	25.9
245	1045	6.8	993	459	1:68	1.0	4.35	271	27.6
250 ³	1734	10.0	952	567	1:57	.75	6.75	234	27.4

¹ See Table VI

² All gauge lengths = 1"

³ Three plating cells

Cell Length: H₂ 13"; #1 37-1/4"; #2 46"; #3 45-1/2"

TABLE III-a

TRIETHYLBORANE-ETHYLENE SYSTEM GAS FLOWS
(SI Units)

Exp. #	Ar	Flow Rates (ml/min)			B:C Ratio	Velocity mps $\times 10^{-3}$	Diameter μ	Avg. U.T.S.* N/cm ² $\times 10^3$	Modulus N/cm ² $\times 10^6$
		Borane	H ₂	C ₂ H ₄					
160	812	12.3	657	369	1:36	2.5	140	230	
161	1262	18.4	997	553	1:36	2.5	178	234	17
162	1262	18.4	997	553	1:36	3.7	156	299	18
164	1262	18.4	997	553	1:36	3.8	156	255	16
167	812	12.3	657	369	1:36	3.8	114	240	18
179	865	12.3	675	369	1:36	3.8	116	261	18
180	836	8.12	634	371	1:52	5.1	95	192	16
181	830	9.15	634	373	1:47	5.1	97	227	18
182	842	10.3	634	371	1:42	5.1	102	192	17
183	843	11.9	657	371	1:37	5.1	107	256	19
186	840	21.8	634	360	1:22	5.1	132	175	17
187	1245	12.3	974	558	1:50	5.1	118	221	16
603	2056	14.3	1581	430	1:36	13.	80	215	
635	1529	10.9	1178	318	1:35	8.1	90	203	18

*All 2.54 cm gauge length

Cell lengths: H₂ 33cm. 1 94.6cm. 2 116.8cm. 3 115.6cm.

TABLE III-b

TRIETHYLBORANE-ETHYLENE SYSTEM GAS FLOWS
(English Units)

Exp. #	Flow Rates (ml/min)				B:C Ratio	Velocity fpm	Diameter mils	Avg. U.T.S.* psi $\times 10^3$	Modulus psi $\times 10^6$
	Ar	Borane	H ₂	C ₂ H ₄					
160	812	12.3	657	369	1:36	.5	5.51	329	
161	1262	18.4	997	553	1:36	.5	7.0	220	24.8
162	1262	18.4	997	553	1:36	.73	6.13	428	26.1
164	1262	18.4	997	553	1:36	.75	6.15	365	23.6
167	812	12.3	657	369	1:36	.75	4.83	344	26
179	865	12.3	675	369	1:36	.75	4.57	374	25.5
180	836	8.12	634	371	1:52	1.0	3.73	275	23.5
181	838	9.5	634	373	1:47	1.0	3.83	325	25.9
182	842	10.3	634	371	1:42	1.0	4.00	275	23.8
183	843	11.9	651	371	1:37	1.0	4.23	367	27.5
186	840	21.8	634	360	1:22	1.0	5.20	251	24.6
187	1245	12.3	974	558	1:50	1.0	4.64	316	22.8
603	2056	14.3	1581	430	1:36	2.56	3.14	308	
635	1529	10.9	1178	318	1:35	1.6	3.56	291	26

*All 1" gauge length

Cell lengths: H₂ 33cm #1 37-1/4"; #2 46"; #3 45-1/2"

TABLE IV

HSiCl₃ - BCl₃ - CH₄ GAS SYSTEM

Exp. #	Velocity		Temperature		Diameter		Texture
	<u>mps × 10⁻³</u>	<u>fpm</u>	<u>(°C)</u>	<u>(°F)</u>	<u>μ</u>	<u>mils</u>	
334	10.7	2.1	1060	1940	36	1.4	Smooth, but weak
335	4.1	.8	1050	1922	43	1.7	Weak, but still smooth
336	4.1	.8	1160	2120	46	1.8	Slightly grainy texture
337	4.1	.8	1200	2192	46	1.8+	Brittle, grainy
338	4.1	.8	1255	2237	54	2.11	Many nodules
339	4.1	.8	1230	2246	55	2.17	Grainy, brittle
340	4.1	.8	1240	2264	52	2.04	Smooth
342	4.1	.8	1100	2072	50	1.98	Smooth, with nodules
343	4.1	.8	1160	2120	58	2.29	Smooth, U.T.S. = 69,800 N/cm ² (100,000 psi)

Cell Length: 94.6 cm (37-1/4")

TABLE V
HEATING CURRENTS AND GAS DISTRIBUTION
BORON TRICHLORIDE-METHANE SYSTEM*

<u>Exp. #</u>	<u>Heating Current (ma)</u>		<u>Plating Cells Gas Flow (%)</u>	
	<u>Plating Cells</u> <u># 1</u>	<u># 2</u>	<u># 1</u>	<u># 2</u>
258	90	110	47.6	52.4
259	84	90	47.6	52.4
273	84	104	43.0	57.0
274	83	102	43.0	57.0
278	83	100	48.2	51.8
279	84	96	45.5	54.5
280	83	93	45.5	54.5

* Refers to experiments in Table I

TABLE VI
HEATING CURRENTS AND GAS DISTRIBUTION
DIBORANE-METHANE SYSTEM*

<u>Exp. #</u>	<u>Heating Current (ma)</u>			<u>Plating Cells Gas Flow (%)</u>		
	<u>Plating Cells</u> <u># 1</u>	<u># 2</u>	<u># 3</u>	<u># 1</u>	<u># 2</u>	<u># 3</u>
208	77	93		35.7	64.3	
209	74	94		36.2	63.8	
211	76	96		44.4	55.6	
214	79	105		44.4	55.6	
220	80	104		44.4	55.6	
230	77	100		36.4	63.6	
234	77	100		36.4	63.6	
237	84	105		37.0	63.0	
245	77	100		36.4	63.6	
250	77	99	145	27.3	36.8	36.8

* Refers to experiments in Table II

TABLE VII

HEATING CURRENTS AND GAS DISTRIBUTION

TRIETHYLBORANE-METHANE SYSTEM*

<u>Exp. #</u>	<u>Heating Current (ma)</u>			<u>Plating Cells Gas Flow (%)</u>		
	<u>Plating Cells</u>			<u># 1</u>	<u># 2</u>	<u># 3</u>
160	84	110				
161	84	110	155	31.8	31.8	36.4
162	84	118	152	32.8	31.1	36.1
164	83	117	150	32.5	30.9	36.6
167	84	112		44.4	55.6	
179	82	101		44.9	55.1	
180	84	104		46.5	53.5	
181	81	103				
182	80	104				
183	81	105				
186	80	106		44.8	55.2	
187	82	106	132	33.3	31.7	35.0
603	82	104	122	28.6	35.7	35.7
635	90	100	106	28.6	35.7	35.7

* Refers to Experiments in Table III

TABLE VIII

EFFECT OF SUBSTRATE LOT CHANGE ON ULTIMATE TENSILE STRENGTH
(SI Units)

<u>Lot #</u>	<u>Diam.</u> <u>μ</u>	<u>Mean U.T.S.</u> <u>$\text{N/cm}^2 \times 10^3$</u>	<u>Std. Dev.</u> <u>$\text{N/cm}^2 \times 10^3$</u>	<u>Coef.</u> <u>of Var.</u>	<u>U.T.S. $\text{N/cm}^2 \times 10^3$</u>
1102	145	392	39	.10	445, 411, 324, 382, 372, 417
1132	143.4	250	22	.09	262, 252, 258, 300, 228

Gauge Length = 2.54 cm; Modulus on order of $18.6 \text{ N/cm}^2 \times 10^6$

(English Units)

<u>Lot #</u>	<u>Diam.</u> <u>mils</u>	<u>Mean U.T.S.</u> <u>$\text{psi} \times 10^3$</u>	<u>Std. Dev.</u> <u>$\text{psi} \times 10^3$</u>	<u>Coef.</u> <u>of Var.</u>	<u>U.T.S. $\text{psi} \times 10^3$</u>
1102	5.71	568	56	.10	645, 596, 470, 554, 540, 605
1132	5.64	377	34	.09	380, 365, 374, 435, 330

Gauge Length = 1"; Modulus on order of $27 \text{ psi} \times 10^6$

TABLE IX

RESISTIVITY OF VARIOUS SUBSTRATE LOTS AT 1400°C IN HYDROGEN

<u>Lot #</u>	<u>Spool</u>	<u>Diameter</u>		<u>E</u>	<u>I</u>	<u>R</u>	<u>ρ</u>
		<u>μ</u>	<u>mils</u>	<u>volts</u>	<u>amps</u>	<u>ohms $\times 10^3$</u>	<u>ohms-inches $\times 10^{-6}$</u>
1070	142	34.0	1.34				
1084	12	33.5	1.32				
1102	10	34.0	1.34	1400	.126	11.29	1.137
				1460	.122	11.97	1.205
1132	3	34.3	1.35	1375	.126	10.91	1.115
1133	4	33.5	1.32	1440	.118	12.20	1.193
1144	1	33.3	1.31	1350	.118	11.44	1.001
1145	1	33.0	1.30	1360	.155	11.83	1.121
Mean						11.61	1.145
Standard Deviation						.44	.040
Coefficient of Variation						.04	.03

TABLE X-a

SELECTED MECH. PROPERTY VS. LOT NO.
FOR 2.54 CM GAUGE LENGTH CARBON SUBSTRATE
(SI UNITS)

<u>Lot No.</u>	<u>Spool</u>	<u>Diameter (μ)</u>	<u>Strain-to-Failure</u>			<u>U.T.S. N/cm² $\times 10^3$</u>			<u>E N/cm² $\times 10^6$</u>		
			<u>Mean</u>	<u>Std.Dev.</u>	<u>Cv.</u>	<u>Mean</u>	<u>Std.Dev.</u>	<u>Cv</u>	<u>Mean</u>	<u>Std.Dev.</u>	<u>Cv</u>
1084 ²	12	33.5	.0228	.0030	.13	90.7	14.5	.16	4.1	.21	.06
1102 ²	10	34.0	.0257	.0040	.15	103.6	17.9	.17	4.1	.21	.06
1132 ²	3	34.3	.0217	.0035	.16	90.7	14.7	.16	4.3	.41	.09
1144 ²	1	33.8	.0230	.0042	.18	93.6	15.9	.17	4.1	.14	.03
1070 ¹	142	34.0	.0209	.0051	.24	84.2	20.7	.25	4.1	.21	.04
1133 ²	4	33.5	.0233	.0038	.16	85.2	11.2	.13	3.9	.34	.09
1145 ³	1	33.0	.0181	.0068	.38	74.3	30.7	.41	4.1	.21	.05

¹ 11 Tests

² 10 Tests

³ 20 Tests

TABLE X-b
 SELECTED MECH. PROPERTY VS. LOT NO.
 FOR 1" GAUGE LENGTH CARBON SUBSTRATE
 (ENGLISH UNITS)

Lot No.	Spool	Diameter (mils)	Strain-to-Failure			U.T.S. psi $\times 10^3$			E psi $\times 10^6$		
			Mean	Std.Dev.	Cv	Mean	Std.Dev.	Cv	Mean	Std.Dev.	Cv.
1084 ²	12	1.32	.0228	.0030	.13	131.6	21	.16	5.9	.3	.06
1102 ²	10	1.34	.0257	.0040	.15	150.3	26	.17	5.9	.3	.06
1132 ²	3	1.35	.0217	.0035	.16	131.6	21.3	.16	6.2	.6	.09
1144 ²	1	1.33	.0230	.0042	.18	135.7	23.1	.17	5.9	.2	.03
1070 ¹	142	1.34	.0209	.0051	.24	122.1	30	.25	5.9	.3	.04
1133 ²	4	1.32	.0233	.0038	.16	123.5	16.2	.13	5.3	.5	.09
1145 ³	1	1.29-1.30	.0181	.0068	.38	107.8	44.5	.41	5.9	.3	.05

¹ 11 Tests

² 10 Tests

³ 20 Tests

TABLE XI
 SELECTED MECHANICAL PROPERTIES VS. LOT NO.
 FOR 25.4 CM (10") GAUGE LENGTH CARBON SUBSTRATE
 (SI Units)

<u>Lot #</u>	<u>Spool</u>	<u>Diameter</u> <u>μ</u>	<u>Strain-to-Failure</u>			<u>U.T.S. N/cm² × 10³</u>			<u>E N/cm² × 10⁶</u>		
			<u>Mean</u>	<u>Std.Dev.</u>	<u>C_v</u>	<u>Mean</u>	<u>Std.Dev.</u>	<u>C_v</u>	<u>Mean</u>	<u>Std.Dev.</u>	<u>C_v</u>
1102 ¹	10	34	.0144	.0011	.08	60.1	5.7	.09	4.27	.07	.02

¹ 10 Tests

(English Units)

<u>Lot #</u>	<u>Spool</u>	<u>Diameter</u> <u>mils</u>	<u>Strain-to-Failure</u>			<u>U.T.S. psi × 10³</u>			<u>E psi × 10⁶</u>		
			<u>Mean</u>	<u>Std.Dev.</u>	<u>C_v</u>	<u>Mean</u>	<u>Std.Dev.</u>	<u>C_v</u>	<u>Mean</u>	<u>Std.Dev.</u>	<u>C_v</u>
1102 ¹	10	1.34	.0144	.0011	.08	87.2	8.2	.09	6.2	.1	.02

¹ 10 Tests

TABLE XII-a
ETCHING ATMOSPHERE FOR CARBON SUBSTRATE
(SI UNITS)

<u>Atmosphere</u>	<u>Velocity M/sec</u>	<u>Temp.. (°C)</u>	<u>Diameter (μ)</u>		<u>Cross-Sectional Area Decrease (%)</u>	<u>Hot Zone Length in cm.</u>
			<u>Initial</u>	<u>Final</u>		
Argon	.0080	1150	32.3	32.3	-0-	.89
		1300	33.3	32.3	3.0	.94
		1410	32.8	32.5	1.5	.89
		1540	32.8	32.3	3.0	.89
		1200	32.8	32.8	-0-	.89
Argon-Oxygen	.0080	1250	33.3	31.2	11.8	
		1110	34.0	31.0	17.1	
		1200	34.3	31.2	16.98	
		1130	33.0	32.3	4.5	
		1110	32.5	32.5	-0-	
Hydrogen	.0080	1310	32.8	31.2	9.1	
		2060	32.8	31.8	6.1	
		2200	32.5	31.2	7.6	
		1675	32.8	30.5	13.5	
		1800	32.8	30.2	14.9	
Hydrogen- Water Vapor	.0080	910	33.3	32.8	3.0	.32
		1000	33.5	31.2	13.1	.32
		1100	33.3	30.7	14.6	.30
		1200	33.8	30.7	17.6	.47
		1300	33.8	29.5	23.9	.64

TABLE XII-b
ETCHING ATMOSPHERES FOR CARBON SUBSTRATES
(ENGLISH UNITS)

<u>Atmosphere</u>	<u>Velocity fpm</u>	<u>Temp. (°F)</u>	<u>Diameter (mils)</u>		<u>Cross-Sectional Area Decrease (%)</u>	<u>Hot Zone Length Inches</u>
Argon	1.58	2102	1.27	1.27	-0-	.35
		2370	1.31	1.29	3.0	.37
		2570	1.29	1.28	1.5	.35
		2804	1.29	1.27	3.0	.35
		2192	1.29	1.29	-0-	.35
Argon-Oxygen	1.58	2282	1.31	1.23	11.8	
		2030	1.34	1.22	17.1	
		2192	1.35	1.23	16.98	
		2066	1.30	1.27	4.5	
		2030	1.28	1.28	-0-	
Hydrogen	1.58	2390	1.29	1.23	9.1	
		3740	1.29	1.25	6.1	
		3992	1.28	1.23	7.6	
		3047	1.29	1.20	13.5	
		3272	1.29	1.19	14.9	
Hydrogen- Water Vapor	1.58	1670	1.31	1.29	3.0	.125
		1832	1.32	1.23	13.1	.125
		2012	1.31	1.21	14.6	.12
		2192	1.33	1.21	17.6	.187
		2372	1.33	1.16	23.9	.25

TABLE XIII

H₂ (H₂O) ETCHING AT 1300°C (2372°F)

<u>Exp. #</u>	<u>Velocity</u>		<u>Initial Diam.</u>		<u>Final Diam.</u>		<u>Hot Zone Length</u>		<u>% Area Decrease</u>
	<u>m/sec</u>	<u>fpm</u>	<u>μ</u>	<u>mils</u>	<u>μ</u>	<u>mils</u>	<u>cm</u>	<u>inches</u>	
43	.0025	0.5	33.0	1.30	24.6	.967			44.7
44	.0051	1.0	33.5	1.32	29.5	1.16	1.14	0.45	22.8
45	.0076	1.49	33.5	1.32	30.2	1.19	1.02	0.40	18.7
46	.0102	2.0	33.3	1.31	32.0	1.26	1.02	0.40	7.5
47	.0127	2.5	33.5	1.32	32.0	1.26	.89	0.35	8.9

TABLE XIV

CALCULATED VS. EXPERIMENTAL ETCHED DIAMETER

<u>Exp. #</u>	<u>Original Diam.</u>		<u>Dwell Time seconds</u>	<u>Experimental Diam.</u>		<u>Calculated Diam.</u>	
	<u>μ</u>	<u>mils</u>		<u>μ</u>	<u>mils</u>	<u>μ</u>	<u>mils</u>
43	33	1.30	4.7455	24.6	0.967	24.5	0.9646
44	33.5	1.32	2.2235	29.5	1.16	29.2	1.1478
45	33.5	1.32	1.394	30.2	1.19	30.7	1.2092
46	33.3	1.31	0.96225	32.0	1.26	31.3	1.2332
47	33.5	1.32	0.710	32.0	1.26	32.1	1.2623

TABLE XV-a

CARBON-BASE MONOFILAMENT PRODUCED
ON ETCHED SUBSTRATE
(SI UNITS)

Run #	Etch Temp °C	Diameter Reduction		Plating Temp ⁴		Tensile ³ N/cm ² × 10 ³	Modulus N/cm ² × 10 ⁶
		Etch <u>μ</u>	Final Dia. <u>μ</u>	<u>1</u>	<u>2</u>		
137 ¹	1350	5.08	79.5	1100	1105	182	17.2
138	1350	4.32	80.3	1230	1100	185	16
139	1370	4.06	80	1220	1200	140	
140	1370	4.06	76.5	1110	1000	228	17.8
144	1460	4.83	75.2	1110	1000	234	19
146 ²	1400	6.10	78	1100	1000	225	18.2
147	1500	7.37	77.7	1100	1000	238	17.9
148	1490	6.35	81.3	1300	960	200	17.4
149	1500	6.86	85.1	1300	1100	141	15.3
150	1500	7.87	82	1300	1200	138	16.3
151	1500	6.6	85.3	1300	1200	180	14.9
142	None		79.5	1105	1000	238	
145	None		81.0	1100	1000	196	15.2

¹ .0087 m/sec

² .0070 m/sec

³ Gauge Lengths = .64 cm

⁴ Cell Lengths: #1 - 94.6 cm; #2 116.8 cm

TABLE XV-b
CARBON-BASE MONOFILAMENT PRODUCED ON ETCHED SUBSTRATE
(ENGLISH UNITS)

Run #	Etch Temp °F	Diameter Reduction		Plating Temp ⁴		Tensile ³ psi × 10 ³	Modulus psi × 10 ⁶
		Etch mils	Final Dia. mils	1	2		
137 ¹	2462	.20	3.13	2032	2021	265	25
138	2462	.17	3.16	2246	2030	268	23.2
139	2498	.16	3.15	2228	2192	200	
140	2498	.16	3.01	2030	1832	228	25.8
144	2660	.19	2.96	2032	1832	234	27.6
146 ²	2552	.24	3.08	2012	1832	327	26.4
147	2732	.29	3.06	2012	1832	345	26
148	2712	.25	3.20	2372	1760	290	25.2
149	2732	.27	3.35	2372	2012	204	22.2
150	2732	.31	3.23	2372	2192	200	23.6
151	2732	.26	3.36	2372	2192	261	21.6
142	None		3.13	2021	1832	345	
145	None		3.19	2012	1832	284	22.1

¹ 1.5 fpm

² 1.2 fpm

³ Gauge Length = 1/4"

⁴ Cell Lengths: #1 - 37-1/4"; #2 - 46"

TABLE XVI

BCl₃ - CH₄ GAS SYSTEM WITH CARBON-PRECOATED SUBSTRATE

Exp. #	Plating Cell Temperature		Diameter		U.T.S.		Appearance
	(°C)	(°F)	(μ)	(mils)	(2.54cm g.l.) (N/cm ² × 10 ³)	(1" g.l.) (psi × 10 ³)	
366	1100	2012	59.69	2.35			Rough, nodules
367			60.96	2.40	192	279	Smooth
369			52.83	2.08	138	200	
370	1100	2012	61.98	2.44	88	128	Smooth
371	1090	1944	60.96	2.40	69	100	
374	1150	2102	65.79	2.59	70	102	Slight grain
375	1210	2210	71.88	2.83	100	145	Grainy

Velocity = .0041 m/sec (0.8 fpm)

Carbon precoat cell temperature = 1550°C (2822°F)

Cell lengths: H₂ 33 cm (13 in); Carbon precoat 94.6 cm (37-1/4 in); #1 116.8 cm (46 in)

TABLE XXVIIa
EFFECT OF BORON CONTENT ON STRENGTH AND MODULUS
(SI Units)

Exp. #	% B	Modulus	U.T.S.	Temperature			Velocity	Diameter	# of Cells	C:B Ratio	Reactive Gas Conc.
		$\text{N/cm}^2 \times 10^6$	$\text{N/cm}^2 \times 10^3$	1	2	3	mps	μ			
162 ¹	33.6	18.0	295	1100	1000	1000	.0037	155.7	3	66:1	20%
168 ¹	34.8	18.6	259	1200	1100	1000	.0037	120.9	3	77:1	20%
186 ¹	35.8	17.0	173	1100	1000		.0051	132.1	2	40:1	20%
187 ¹	31.0	15.7	217	1100	1000	1000	.0051	117.9	3	100:1	20%
196 ¹	32.1	17.6	139	1100	1000		.0051	109.5	2	80:1	30%
197 ¹	32.1	17.0	173	1100	1000		.0051	105.4	2	90:1	30%
208 ²	37.1	21.2	223	1100	1000		.0038	125.7	2	50:1	20%
211 ²	33.0	17.7	124	1100	1000		.0040	102.1	2	100:1	20%
219 ²	34.5	16.1	132	1100	1000		.0038	123.3	2	84:1	20%
220 ²	36.2	18.1	179	1150	1000		.0038	137.2	2	40:1	20%

¹ Organoborane - ethylene

² Boron Hydride - ethylene

U.T.S. measured on 2.54 cm gauge length

TABLE XXVIIb
EFFECT OF BORON CONTENT ON STRENGTH AND MODULUS
(English Units)

Exp. #	% B	Modulus psi $\times 10^6$	U.T.S. psi $\times 10^3$	Temperature			Velocity fpm	Diameter mils	# of Cells	C:B Ratio	Reactive Gas Conc.
				1	2	3					
162	33.6	26.1	428	2012	1832	1832	0.73	6.13	3	66:1	20%
168	34.8	27.0	375	2192	2012	1832	0.73	4.76	3	77:1	20%
186	35.8	24.6	251	2012	1832		1.0	5.20	2	40:1	20%
187	31.0	22.8	315	2012	1832	1832	1.0	4.64	3	100:1	20%
196	32.1	25.5	201	2012	1832		1.0	4.31	2	80:1	30%
208	37.1	30.75	324	2012	1832		0.74	4.95	2	50:1	20%
211	33.0	25.6	180	2012	1832		0.78	4.02	2	100:1	20%
219	34.5	23.4	191	2012	1832		0.74	4.85	2	84:1	20%
220	36.2	26.2	260	2102	1832		0.74	5.40	2	40:1	20%

¹ Organoborane - ethylene

² Boron Hydride - ethylene

U.T.S. measured on 1" gauge length

TABLE XVIII

ALLOY DEPOSITION ON TUNGSTEN SUBSTRATE
(SI Units)

<u>Exp. #</u>	<u>Substrate</u> <u>μ</u>	<u>Temperature (°C)</u>			<u>Velocity</u> <u>m/sec</u>	<u>Diameter</u> <u>μ</u>	<u>U.T.S.</u> <u>N/cm² × 10³</u>
		<u>1</u>	<u>2</u>	<u>3</u>			
288	25.4	1240			.0041	72.9	69
290	12.7	1240	1140		.0041	93.5	69
292	12.7	1130	1020		.0081	71.1	81.4, 128.2
294	12.7	1088	980		.0081	66.0	69
295	25.4	1240	1000	1000	.0081	102.9	99.3, 94.5
296	25.4	1100	990		.0081	95.8	128, 122
298	12.7	1060	990		.0041	87.6	69
299	12.7	1050	990		.0081	72.1	110.3, 120.7

Gauge Lengths = 2.54 cm

Cell Lengths: #1 - 94.6 cm; #2 - 116.8 cm; #3 - 115.6 cm

(English Units)

<u>Exp. #</u>	<u>Substrate</u> <u>mils</u>	<u>Temperature (°F)</u>			<u>Velocity</u> <u>fpm</u>	<u>Diameter</u> <u>mils</u>	<u>U.T.S.</u> <u>psi × 10³</u>
		<u>1</u>	<u>2</u>	<u>3</u>			
288	1.0	2264			0.8	2.87	100
290	0.5	2264	2084		0.8	3.68	100
292	0.5	2066	868		1.6	2.80	118, 186
294	0.5	1958	1796		1.6	2.60	100
295	1.0	2264	1832	1832	1.6	4.05	144, 137
296	1.0	2012	1814		1.6	3.77	186, 177
298	0.5	1940	1814		0.8	3.45	100
299	0.5	1922	1814		1.6	2.84	160, 175

Gauge Lengths = 1"

Cell Lengths: #1 - 37-1/4"; #2 - 46"; #3 - 45-1/2"

TABLE XIX-a
SIMULTANEOUS TEMPERATURE CHANGES FOR EACH PLATING CELL
(SI Units)

Exp. No.	(°C) Plating Cell Temperatures				Diameter μ	Avg. U.T.S.* $\text{N/cm}^2 \times 10^3$
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>		
78	1200	1090	1090	1080	106.4	186.2
79	1400	1300	1300	1300	105.9	66.2
80	1350	1250	1250	1250	103.4	169.6
81	1300	1200	1210	1200	99.8	104.7
82	1280	1150	1150	1145	104.6	206.2
83	1150	1050	1050	1050	103.9	166.7
84	1100	1000	1000	1000	101.1	293
85	1050	950	950	950	104.6	139.3
88	1100	1000	1050	1025	99.8	165.5
89	1100	1000	1020	1010	100.1	259.9

* Gauge Length: .635cm

Velocity: .0123mps

Cell Length: #1 94.6cm; #2 116.8cm; #3 115.7cm; #4 111.8cm;

H₂ Cell 33cm 1350°C

Flow Rates (ml/min) Ar 2095

H₂ 1580

TEB 14.5

C₂H₄ 423

C:B Ratio 65;1

% Reactive 10

% Distribution 1: 20.65

2: 27.17

3: 26.09

4: 26.09

TABLE XIX-b

SIMULTANEOUS TEMPERATURE CHANGES FOR EACH PLATING CELL
(English Units)

Exp. #	Plating Cell Temperatures (°F)				Diameter mils	Avg. U.T.S.* psi × 10 ³
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>		
78	2192	1994	1994	1976	4.19	270
79	2552	2372	2372	2372	4.17	96
80	2462	2280	2280	2280	4.07	246
81	2372	2192	2210	2192	3.93	204
82	2336	2102	2102	2093	4.12	299
83	2102	1922	1922	1922	4.09	242
84	2012	1832	1832	1832	3.98	425
85	2012	1742	1742	1742	4.12	202
88	1922	1832	1922	1877	3.93	240
89	2012	1832	1868	1850	3.94	377

* Gauge Length = 1/4"

Velocity = 2.43 fpm

Cell Lengths: #1 - 37-1/4"; #2 - 46"; #3 - 45-1/2"; #4 - 49"

H₂ Cleaning Cell = 13", 2462°F

Flow Rates (ml/min) Ar 2095

H₂ 1580

TEB 14.5

C₂H₄ 423

C:B Ratio 65:1

% Reactive 10

% Distribution #1: 20.65

#2: 27.17

#3: 26.09

#4: 26.09

TABLE XX
EFFECTS OF VARYING FIRST PLATING CELL TEMPERATURE
(SI Units)

Exp. #	1st Cell* Temp (°C)	Diameter μ	Modulus $\text{N/cm}^2 \times 10^6$	U.T.S. - $\text{N/cm}^2 \times 10^3$	
				.64 cm g.l.	2.54 cm g.l.
126	1100	99.8		228, 232	
127	1000	97.0	14.2	181, 181, 194	124, 127
128	1050	97.8	10.7	160, 185, 163	206, 207
129	1150	99.1	15.3	210, 202, 237	95, 200
130	1210	100.3	13.9	138, 186, 226	217, 151
131	1260	100.6	14.5	157, 216, 208	143, 103
132	1350	100.1	14.8	209, 186, 178	88, 111
133	1350	102.4	12.9	123, 132, 188	108
134	1400	103.1	12.76	103, 145	
135	1100	96.8	18.6	212, 233, 175	154, 157

* All other plating cells at nominally 1000°C

All velocities at .0123 mps

Cell Lengths: H₂- 33 cm; #1- 94.6 cm; #2- 116.8 cm; #3- 115.6 cm; #4- 111.8 cm

(English Units)

Exp. #	1st Cell* Temp (°F)	Diameter mils	Modulus $\text{psi} \times 10^6$	U.T.S. - $\text{psi} \times 10^3$	
				1/4" g.l.	1" g.l.
126	2012	3.93		331, 336	
127	1832	3.82	20.6	262, 262, 282	180, 184
128	1922	3.85	15.5	232, 268, 236	299, 300
129	2102	3.90	22.2	305, 293, 343	138, 290
130	2210	3.95	20.2	200, 270, 328	314, 219
131	2300	3.96	21.0	228, 313, 301	207, 150
132	2462	3.94	21.4	303, 270, 328	128, 161
133	2462	4.03	18.7	178, 192, 272	158
134	2552	4.06	18.5	150, 210	
135	2012	3.81	27.0	308, 338, 254	224, 228

* All other plating cells at nominally 1832°F

All velocities at 2.43 fpm

Cell Lengths: H₂- 13"; #1- 37-1/4"; #2- 46"; #3- 45-1/2"; #4- 44"

Flow rates (ml/min): Ar 2095; H₂ 1580; Triethyl 14.5; C₂H₄ 427

% Distribution: #1- 20.74; #2- 26.6; #3- 26.1; #4- 26.6

TABLE XXI
 REACTIVE GAS CONCENTRATION TRIETHYLBORANE-ETHYLENE SYSTEM
 (SI Units)

Exp #	Temp (°C) by Cell			Dia. (μ)	$\frac{E}{N/cm^2} \times 10^6$	Plating Gas Conc (%)	Tensile Strength $N/cm^2 \times 10^3$		Flow Rates (ml/min)				C:B Ratio	% Distribution		
	1	2	3				.64cm g.l.	2.54cm g.l.	Ar	H ₂	TEB	C ₂ H ₄		1	2	3
153	1120	990		93.73	16	20	218, 221, 272	245, 259, 290	852	655	12.4	363	65:1	42.6	57.5	
154	1100	1000	1000	116.59	14	20	213, 145, 169	172, 204, 148	1283	944	18.4	544	65:1	32.3	30.6	37.1
155	1130	1010		91.69	18	20	294, 277	197, 219	852	655	12.4	363	65:1	42.6	57.5	
157	1110	990		99.82	15	30	278, 243, 203	148, 228	750	565	18.6	545	65:1	42.6	57.5	
158a	1105	1005	1000	101.6	15	10	170, 171, 312		1446	1084	9.8	292	65:1	28.6	35.7	35.7

Velocity = .0076 mps

Cell length: H₂ 33 cm; #1 94.6 cm; #2 116.8 cm; #3 115.7 cm

(English Units)

Exp #	Temp (°F) by Cell			Dia. (mils)	$\frac{E}{psi} \times 10^6$	Plating Gas Conc (%)	Tensile Strength $psi \times 10^3$		Flow Rates (ml/min)				C:B Ratio	% Distribution		
	1	2	3				1/4" g.l.	1" g.l.	Ar	H ₂	TEB	C ₂ H ₄		1	2	3
153	2048	1814		3.69	23.4	20	316, 221, 394	355, 375, 420	852	655	12.4	363	65:1	42.6	57.5	
154	2012	1832	1832	4.59	24.1	20	309, 210, 245	250, 298, 214	1283	944	18.4	544	65:1	32.3	30.6	37.1
155	2066	1850		3.61	26.0	20	427, 402	285, 315	852	655	12.4	363	65:1	42.6	57.5	
157	2030	1814		3.93	22.0	30	403, 353, 295	214, 330	750	565	18.6	545	65:1	42.6	57.5	
158a	2021	1841	1832	4.0	21.4	10	246, 248, 452		1446	1084	9.8	292	65:1	28.6	35.7	35.7

Velocity = 1.5 fpm

Cell length: H₂ 13"; #1 37-1/4"; #2 46"; #3 45-1/2"

TABLE XXII-a
CARBON TO BORON RATIOS FOR TRIETHYLBORANE-ETHYLENE
(SI UNITS)

Exp. No.	C:B Ratio	Dia, (μ)	E $\text{N/cm}^2 \times 10^6$	Tensile Strength ¹ $\text{N/cm}^2 \times 10^3$	Ar	Flow Rates (ml/min)			Distribution		
						H ₂	C ₂ H ₄	TEB	1	2	3
167	65.5:1	122.68	19.9	187, 268, 256	817	654	368	12.4	44.9	55.1	
168	65.4:1	120.90	18.6	262, 209, 273	841	659	370	12.5	44.9	55.1	
170	65.5:1	161.54	15.9	121, 170, 143	1263	993	556	18.7	32.8	36.1	31.2
178	65.5:1	117.35	18.5	250, 185, 197	817	654	368	12.4	44.9	55.1	
179	65.5:1	116.08	17.6	236, 279	817	654	368	12.4	44.9	55.1	
183 ²	67.4:1	108.71	19	332, 230, 196	846	645	370	12.0			
198	58.0:1	128.78	19	205, 171, 174	846	632	367	14.2	49.4	50.6	
199	64.0:1	122.17	17.6	197, 197, 95	847	645	368	12.8			
200	61.0:1	127.00	17.6	204, 159, 279	910	654	368	13.4	45.5	54.5	
201	65.0:1	122.68	16.8	183, 187, 196	847	645	368	12.4	45.5	54.5	
202	64.0:1	154.43	15.2	280, 237, 179	1278	970	556	19.2	33.3	33.3	33.3
203	58.0:1	164.34	17.9	161, 167, 174	1263	993	550	21.1	33.3	33.3	33.3

¹ 2.54cm Gauge Length

² Velocity - .0051mps (all others .0038 mps)

Cell Length: H₂ 33 cm; #1 94.6 cm; #2 116.8 cm; #3 115.7 cm

All 20% reactive

TABLE XXII-b
CARBON TO BORON RATIOS FOR TRIETHYLBORANE- ETHYLENE
(ENGLISH UNITS)

Exp. No.	C:B Ratio	Dia. (mils)	E (psi $\times 10^6$)	Tensile Strength (psi $\times 10^3$ 1" g.l.)	Ar	Flow Rates (ml/min)			Distribution		
						H ₂	C ₂ H ₄	TEB	1	2	3
167	65.5:1	4.83	26.0	271, 388, 372	817	654	368	12.4	44.9	55.1	
168	65.4:1	4.76	27.0	380, 303, 396	841	659	370	12.5	44.9	55.1	
170	65.5:1	6.36	23.0	175, 246, 208	1263	993	556	18.7	32.8	36.1	31.2
178	65.5:1	4.62	26.9	362, 268, 286	817	654	368	12.4	44.9	55.1	
179	65.5:1	4.57	25.5	342, 405	817	654	368	12.4	44.9	55.1	
183*	67.4:1	4.28	27.5	482, 334, 284	846	645	370	12.0			
198	58.0:1	5.07	27.5	298, 248, 252	846	632	367	14.2	49.4	50.6	
199	64.0:1	4.81	25.5	285, 285, 138	847	645	368	12.8			
200	61.0:1	5.0	25.5	296, 230, 405	910	654	368	13.4	45.5	54.5	
201	65.0:1	4.83	24.4	265, 271, 284	847	645	368	12.4	45.5	54.5	
202	64.0:1	6.08	22.0	406, 344, 259	1278	970	556	19.2	33.3	33.3	33.3
203	58.0:1	6.47	25.9	234, 242, 253	1263	993	550	21.1	33.3	33.3	33.3

* Velocity - 1 fpm (all others 0.75)

Cell Length: H₂ 13"; #1 37-1/4"; #2 46"; #3 45-1/2"

All 20% reactive

TABLE XXIII-a
ADDITIONAL CARBON TO BORON RATIOS FOR TRIETHYLBORANE-ETHYLENE
(SI UNITS)

Exp. #	C:B Ratio	Dia. μ	E $N/cm^2 \times 10^6$	Tensile Strength $N/cm^2 \times 10^3$	Ar	Flow Rates (ml/min)			Distribution		
						H ₂	C ₂ H ₄	TEB	1	2	3
180	96.0:1	94.74	16.2	172,	842	645	370	8.2	46.5	53.5	
181	87.0:1	97.28	17.9	274, 167, 231	845	645	374	9.2	46.5	53.5	
182	77.0:1	121.92	16.4	238, 194, 138	846	645	370	10.4	46.5	53.5	
184	58.0:1	111.00	14.5	276, 376, 88	846	645	367	14.2	46.5	53.5	
185	48.0:1	117.09	16.4	279, 216, 177	849	645	364	17.3	46.5	53.5	
186	38.0:1	132.08	17.0	246, 151, 126	847	645	359	22.1	44.8	55.2	
187	96.0:1	117.86	15.7	264, 181, 208	1264	970	556	12.4	33.6	31.9	34.5
188	87.0:1	123.70	16.3	308, 208, 230, 129	1271	968	559	13.8	33.6	31.9	34.5
189	77.0:1	130.05	15.7	208, 228, 150	1270	968	557	15.7	33.6	31.9	34.5
190	67.0:1	136.65	16.5	228, 208, 150	1270	968	554	18.1	33.6	31.9	34.5
191	58.0:1	142.24	15.4	183, 179, 149	1271	968	551	21.3	33.6	31.9	34.5
196*	77.0:1	109.47	17.6	225, 152, 171	741	564	556	15.6	50.0	50.0	

* at 30% Reactive gas concentration ; all others 20%

Velocity - .0051 mps

Gauge Length=2.54cm.

TABLE XXIII-b
ADDITIONAL CARBON TO BORON RATIOS FOR TRIETHYLBORANE-ETHYLENE

Exp. No.	C:B Ratio	Dia. mils	E	Tensile Strength	Ar	Flow Rates (ml/min)			Distribution		
			psi $\times 10^6$	psi $\times 10^3$		H ₂	C ₂ H ₄	TEB	1	2	3
180	96.0:1	3.73	23.5	250, 346, 229	842	645	370	8.2	46.5	53.5	
181	87.0:1	3.83	25.9	398, 242, 335	845	645	374	9.2	46.5	53.5	
182	77.0:1	4.80	23.8	345, 282, 200	846	645	370	10.4	46.5	53.5	
184	58.0:1	4.37	21.0	400, 545, 127	846	645	367	14.2	46.5	53.5	
185	48.0:1	4.61	23.8	405, 313, 257	849	645	364	17.3	46.5	53.5	
186	38.0:1	5.20	24.6	357, 219, 183	847	645	359	22.1	44.8	55.2	
187	96.0:1	4.64	22.8	383, 262, 302	1264	970	556	12.4	33.6	31.9	34.5
188	87.0:1	4.87	23.7	446, 302, 333, 187	1271	968	559	13.8	33.6	31.9	34.5
189	77.0:1	5.12	22.8	302, 331, 218	1270	968	557	15.7	33.6	31.9	34.5
190	67.0:1	5.38	24.0	330, 302, 218	1270	968	554	18.1	33.6	31.9	34.5
191	58.0:1	5.60	22.4	265, 260, 216	1271	968	551	21.3	33.6	31.9	34.5
196*	77.0:1	4.31	25.5	326, 220, 248	741	564	556	15.6	50.0	50.0	

* at 30% Reactive gas concentration

Velocity - 1 fpm

Cell Length: H₂ - 13"; #1 37-1/4"; #2 46"; #3 45-1/2"

Gauge Length = 1"

TABLE XXIV-a
CARBON TO BORON RATIOS FOR DIBORANE-ETHYLENE
(SI UNITS)

Exp. No.	C:B Ratio	Diameter (μ)	Modulus ($\text{N/cm}^2 \times 10^6$)	Tensile Strength ($\text{N/cm}^2 \times 10^3$ -2.54 cm.)	Ar	Flow Rates (ml/min)			Distribution	
						H ₂	C ₂ H ₄	Diborane	1	2
208	51	125.73	21.2	223, 241, 205	1205	645	377	7.3	35.7	64.3
209	51	130.81	20.0	276, 177, 212	1265	546	377	7.3	36.2	63.8
211	101	102.11	17.7	100, 99, 174	851	654	377	3.7	44.4	55.6
212	92	105.66	16.3	203, 181, 163	949	541	378	4.1	44.4	55.6
213	82	109.73	17.9	146, 165, 170	952	519	376	4.6	44.4	55.6
214	72	116.08	18.8	145, 84, 222	970	496	376	5.2	44.4	55.6
216	51	128.78	17.0	183, 252, 174	1196	645	370	7.3	44.4	55.6
219	85	123.19	16.1	76, 128, 191	996	496	522	6.1	34.5	65.5
220	40.6	137.16	18.1	178, 174, 185	1073	406	370	9.1	46.4	55.6
224	61	122.68	18.2	124, 192, 172	848	645	374	6.1	46.4	55.6
225	45.8	130.81	18.8	189, 174, 162	849	645	372	8.1	46.4	55.6
228	76	114.30	15.7	152, 145, 108	845	631	376	4.9	46.4	55.6
229	50	128.52	20.4	245, 156, 197	1196	654	370	7.3	46.4	55.6
230	54	127.25	20.3	259, 252, 124, 322	1151	632	370	6.8	46.4	55.6
232		127.51	19.4	316, 314, 269					35.7	64.3
233		127.00	19.9	201, 248, 324					35.7	64.3

All two chamber runs; all at 20% Reactive gas concentration; Velocity = .005 mps

Stage 2 at 1100°C; Stage 3 at 1000°C

Cell lengths: #1 94.6cm; #2 116.8cm

H₂ Cell 33cm 1350°C

TABLE XXIV-b
CARBON TO BORON RATIOS FOR DIBORANE-ETHYLENE
(ENGLISH UNITS)

Exp. No.	C:B Ratio	Diameter (mils)	Modulus (psi $\times 10^6$)	Tensile Strength (psi $\times 10^3$ -1" g.l.)	Ar	Flow Rates (ml/min)			Distribution	
						H ₂	C ₂ H ₄	Diborane	1	2
208	51	4.95	30.1	324, 250, 297	1205	645	377	7.3	35.7	64.3
209	51	5.15	29.0	400, 257, 308	1265	546	377	7.3	36.2	63.8
211	101	4.02	25.6	145, 143, 253	851	654	377	3.7	44.4	55.6
212	92	4.16	23.7	294, 262, 236	949	541	378	4.1	44.4	55.6
213	82	4.32	26.0	212, 239, 246	952	519	376	4.6	44.4	55.6
214	72	4.57	27.2	210, 122, 322	970	496	376	5.2	44.4	55.6
216	51	5.07	24.7	265, 365, 253	1196	645	370	7.3	44.4	55.6
219	85	4.85	23.4	110, 186, 277	996	496	522	6.1	34.5	6.55
220	40.6	5.40	26.2	258, 253, 268	1073	406	370	9.1	46.4	55.6
224	61	4.83	26.4	180, 279, 249	848	645	374	6.1	46.4	55.6
225	45.8	5.15	27.2	274, 253, 235	849	645	372	8.1	46.4	55.6
228	76	4.50	22.7	220, 302, 157	845	631	376	4.9	46.4	55.6
229	50	5.00	29.6	210, 226, 285	1196	654	370	7.3	46.4	55.6
230	54	5.01	29.4	376, 366, 180, 467	1151	632	370	6.8	46.4	55.6
232		5.02	28.1	316, 314, 269					35.7	64.3
233		5.0	28.8	292, 359, 470					35.7	64.3

All two chamber runs; all at 20% Reactive gas Concentration; velocity = 1 fpm

Stage 2 at 2012°F; Stage 3 at 1832°F

Cell lengths: H₂ 13"; #1 37-1/4"; #2 46"

TABLE XXV-a

C:B RATIO AND VELOCITY EFFECTS FOR DIBORANE- ETHYLENE
(SI UNITS)

Exp. No.	C:B Ratio	Vel. (mps)	Dia. (μ)	E $N/cm^2 \times 10^6$	U.T.S.* $N/cm^2 \times 10^3$	Flow Rates (ml/min)				% Distribution		
						Ar	H ₂	C ₂ H ₄	Diborane	1	2	3
234	54:1	.0051	114.05	19.0	188, 188, 197	1151	632	370	6.8	36.4	63.6	
236	54:1	.0025	150.37	21.6	196, 145, 82	1151	632	370	6.8	36.4	63.6	
237	54:1	.0076	100.84	17.9	174, 157, 103	1151	632	370	6.8	36.4	63.6	
239A	46:1	.0070	108.20	17.4	160, 77, 62	1277	632	370	8.1	44.7	55.3	
239B	67:1	.0051	107.95	17.4	234, 200, 76	846	632	374	5.6	44.7	55.3	
240	67:1	.0025	155.70	18.5	171, 128, 96	846	632	374	5.6	44.7	55.3	
241	46:1	.0051	124.21	19.0	208, 169, 191	1277	632	370	8.1	44.7	55.3	
242	46:1	.0025	161.54	17.9	152, 143, 109	1277	632	370	8.1	44.7	55.3	
243	66:1	.0071	95.25	14.7	188, 79, 41	855	654	376	5.7	44.7	55.3	
244	54:1	.0038	127.00	19.3	227, 172, 248	1151	632	370	6.8	36.4	63.6	
245	66:1	.0051	110.49	19.0	232, 167, 161	1044	790	457	6.8	36.4	63.6	
246	94:1	.0038	140.21	17.2	152, 113, 88	1337	948	556	5.9	28.8	36.4	34.8
247	53:1	.0038	177.04	16.3	149, 83, 54	1825	993	578	11.0	27.2	37.0	35.8
248	75:1	.0038	153.42	16.5	160, 152, 124	1357	993	565	7.5	31.2	34.4	34.4
249	58:1	.0038	169.42	15.4	128, 121, 99	1319	1015	556	9.5	31.2	34.4	34.4
250	55:1	.0038	171.45	18.9	170, 159, 156	1753	948	565	10.2	26.4	36.8	36.8
251	54:1	.0038	179.32	17.9	130, 119, 81	2093	1148	674	12.4	22.5	38.2	39.3
252	81:1	.0038	153.42	17.0	186, 169, 124	1491	812	587	7.2	31.2	34.4	34.4
253	76:1	.0039	155.42	17.0	174, 164, 82	1312	970	587	7.7	31.2	34.4	34.4
254	66:1	.0038	162.31	16.3	135, 121, 114	1430	1015	587	8.9	31.2	34.4	34.4
255	54:1	.0038	132.33	18.9	243, 232, 185, 193	1151	632	370	6.8	36.4	63.6	
256	54:1	.0038	130.05	19.7	215, 179, 165	1151	632	370	6.8	36.4	63.6	

* Gauge Lengths - 2.54 cm.

All at 20% Reactive gas concentration

First Plating Cell at 1100°C; all others nominally 1000°C

Cell lengths: H₂ 33cm; #1 94.6cm; #2 116.8cm; #3 115.7cm

TABLE XXV-h
C:B RATIO AND VELOCITY EFFECTS FOR DIBORANE-ETHYLENE
(ENGLISH UNITS)

Exp. No.	C:B Ratio	Vel. (fpm)	Dia. (mils)	E (psi $\times 10^5$)	U.T.S. (psi $\times 10^3$)	Flow Rates (ml/min)				Distribution		
						Ar	H ₂	C ₂ H ₄	Diborane	1	2	3
234	54:1	1.0	4.49	27.6	237, 237, 286	1151	632	370	6.8	36.4	63.6	
236	54:1	0.5	5.92	31.3	284, 210, 119	1151	632	370	6.8	36.4	63.6	
237	54:1	1.5	3.97	25.9	252, 228, 250	1151	632	370	6.8	36.4	63.6	
239A	46:1	1.37	4.26	25.2	232, 112, 90	1277	632	370	8.1	44.7	55.3	
239B	67:1	1.0	4.25	25.2	339, 290, 110	846	632	374	5.6	44.7	55.3	
240	67:1	0.5	6.13	26.9	248, 186, 139	86	632	374	5.6	44.7	55.3	
241	46:1	1.0	4.89	27.5	301, 245, 277	1277	632	370	8.1	44.7	55.3	
242	46:1	0.5	6.36	25.9	220, 207, 158	1277	632	370	8.1	44.7	55.3	
243	66:1	1.4	3.75	21.3	273, 114, 60	855	654	376	5.7	44.7	55.3	
244	54:1	0.74	5.0	28.0	329, 250, 214	1151	632	370	6.8	36.4	63.6	
245	66:1	1.0	4.35	27.6	336, 242, 234	1044	790	457	6.8	36.4	63.6	
246	94:1	0.75	5.52	25.0	221, 164, 127	1337	948	556	5.9	28.8	36.4	34.8
247	53:1	0.75	6.97	23.6	216, 120, 78	1825	993	578	11.0	27.2	37.0	35.8
248	75:1	0.75	6.04	24.0	232, 220, 180	1357	993	565	7.5	31.2	34.4	34.4
249	58:1	0.75	6.67	22.3	186, 175, 143	1319	1015	556	9.5	31.2	34.4	34.4
250	55:1	0.75	6.75	27.4	246, 230, 226	1753	948	565	10.2	26.4	36.8	36.8
251	54:1	0.75	7.06	26.0	189, 172, 118	2093	1148	674	12.4	22.5	38.2	39.3
252	81:1	0.75	6.04	24.7	270, 245, 180	1491	812	587	7.2	31.2	34.4	34.4
253	76:1	0.77	6.12	25.7	252, 238, 119	1312	970	587	7.7	31.2	34.4	34.4
254	66:1	0.75	6.39	23.6	196, 175, 165	1430	1015	587	8.9	31.2	34.4	34.4
255	54:1	0.75	5.21	27.4	352, 336, 269, 280	1151	632	370	6.8	36.4	63.6	
256	54:1	0.74	5.12	28.5	312, 259, 239	1151	632	370	6.8	36.4	63.6	

All at 20% Reactive gas concentration

First Plating Cell at 2012°F; all others nominally 1832°F

Cell Lengths: H₂ 13"; #1 37-1/4"; #2 46"; #3 45-1/2"

Gauge Lengths = 1"

TABLE XXVI
BORON HALIDE - HYDROCARBON GAS SYSTEM^a
(SI Units)

Exp. No.	C:B Ratio	Dia. (μ)	Temperature ($^{\circ}$ C)		Modulus (N/cm ² \times 10 ⁶)	U.T.S. ⁴ (N/cm ² \times 10 ³)
			<u>1</u>	<u>2</u>		
258	1:2.4	144.3	1400	Vibration	26.9	241, 243, 194
259 ¹	1:2.4	112.5	1350	1180	32.4	269, 282, 292
271 ²		121.2	1300	1210	22.1	179, 159
273 ²	2.7:1	133.6	1310	1220	24.8	226, 194, 81
274 ²	3.3:1	139.7	1300	1220	26.3	150, 136
275 ²		143.5	1350	1200	26.9	161, 141, 138
276		134.6	1350	1180	29.6	ca 69
278	1:1.3	129.3	1350	1180	25.9	193, 225, 204
279 ³	1:2.4	117.3	1340	1180	29.0	204, 123, 91
280 ³	1:2.4	115.8	1360	1180	30.0	136, 115

¹ Unable to repeat this

² BCL₃ + C₂H₄ others BCL₃ & CH₄

³ Velocity .0043 mps (all others at .0038 mps)

^a Flow Rates shown on Table 1

Cell Lengths: H₂ 33cm; #1 94.6cm; #2 116.8cm

⁴ Gauge Length = 2.54cm

(English Units)

Exp. No.	C:B Ratio	Dia. (mils)	Temperature ($^{\circ}$ F)		Modulus (psi \times 10 ⁶)	U.T.S. ⁴ (psi \times 10 ³)
			<u>1</u>	<u>2</u>		
258	1:2.4	5.68	2552	Vibration	39.0	350, 340, 281
259 ¹	1:2.4	4.43	2462	2156	47.0	390, 410, 423
271 ²		4.77	2372	2210	32.0	260, 231
273 ²	2.7:1	5.26	2390	2228	35.9	328, 282, 227
274 ²	3.3:1	5.50	2370	2228	38.2	217, 198
275 ²		5.65	2462	2192	39.0	234, 204, 200
276		5.30	2462	2156	43.0	ca 100
278	1:1.3	5.09	2462	2156	37.5	280, 326, 296
279 ³	1:2.4	4.62	2444	2156	42.0	296, 178, 132
280 ³	1:2.4	4.56	2480	2156	43.5	197, 167

¹ Unable to repeat this

² BCL₃ + C₂H₄ others BCL₃ & CH₄

³ Velocity 0.85 fpm (all others at 0.75 fpm)

⁴ Gauge Length = 1"

Cell lengths: H₂ 13"; #1 37-1/4"; #2 46"

TABLE XVII

TRIETHYLBORANE-ETHYLENE GAS SYSTEM WITH PRECOATED SUBSTRATE

Exp. #	Plating Cell Temperature		C:B Ratio	Diameter		U.T.S. - 2.54 cm (1") g.l.	
	$^{\circ}\text{C}$	$^{\circ}\text{F}$		μ	mils	$\text{N/cm}^2 \times 10^3$	$\text{psi} \times 10^3$
376	1150	2102	128:1	71.88	2.83	99	143
377	1200	2192	128:1	73.15	2.88	135, 152	196, 220
378	1250	2282	128:1	72.39	2.85	154	223
379	1160	2120	221:1	61.21	2.41	138	200
381	1100	2012	165:1	68.58	2.70	156	227
382	1100	2012	86:1	81.79	3.22	256	372
383	1100	2012	46:1	99.57	3.92	381	552
384	1100	2012	29:1	125.73	4.95	308, 167, 283	446, 242, 410
385	1050	1922	21:1	100.33	3.95		
390	¹	¹	46:1	100.33	3.95	303, 303	440, 440
391	¹	¹	46:1	95.76	3.77	229, 322	332, 467
392	¹	¹	46:1	96.76-94.45	3.77-3.72	286 ²	415 ²
393	¹	¹	46:1	95.25	3.75	472, 214, 266	685, 310, 386
399	¹	¹	46:1	97.28	3.83	183, 192, 244	265, 278, 354
400	¹	¹	46:1	97.79	3.85	246 ³	357 ³
410	1110	2030	46:1	99.31	3.91	251, 180, 159, 103, 83	364, 261, 230, 150, 120

¹ Plating chamber became too dark for temperature readings

² Average of 28 tests taken over a 21.4 meters (70 feet) span, coefficient of variation = 18%

³ Average over a 14.7 meters (50 feet) span

Cell Lengths: Pre-coat cell - 94.6 cm (37-1/4") (1550°C); Plating cell - 116.8 cm (46")

TABLE XXVIII

BEST PROPERTY COMBINATIONS IN ONE MONOFILAMENT

<u>Contract No.</u>	<u>Diameter</u>		<u>U.T.S. (Exp. high)</u>		<u>Modulus</u>	
	<u>μ</u>	<u>(mils)</u>	<u>N/cm^2</u>	<u>psi</u>	<u>$N/cm^2 \times 10^5$</u>	<u>psi $\times 10^6$</u>
NAS3-12429	83.8	3.3	373,000	541,000 ¹	18.5	27
NAS3-14325	87.	3.44	381,000	552,000 ¹	18.5	27
This effort	145.	5.71	451,000	645,000 ²	18.5	27

¹ .64 cm (1/4 in) gauge length

² 2.54 cm (1 in) gauge length

TABLE XXIX

PROJECTED COSTS FOR LARGE DIAMETER CARBON-ALLOY MONOFILAMENT
 (based on 5.6 mil diameter fiber, ca. 35% boron content)
 (30,000 to 40,000 lbs. per year)

<u>Raw Material</u>	<u>Amount Required</u>	<u>Cost/unit</u>	<u>Total</u>
Triethyl borane	10.78 lbs	\$2.00 ¹	\$21.56
Ethylene	81.09 lbs	.04 ²	3.24
Hydrogen	9.41 lbs	.60 ³	5.65
Argon	240.2 lbs	.052 ³	12.49
Carbon substrate	.0345 lbs (34,500 feet)	230.00 ⁴	7.94
Electric power (assume power factor = 0.83)	719. kwhr	.02	14.28
			<hr/> \$65.16
Estimated overhead (includes labor, profit, etc.) 50%			32.58
			<hr/>
TOTAL COST/POUND			\$97.74

¹ A. J. Toering, Callery Chemical Co., private communication, March 21, 1973

² O. K. Dowd, Phillips Petroleum Co., private communication, April 6, 1973

³ C. Droege, Union Carbide Corp., Linde Div., private communication, April 23, 1973

⁴ W. R. Benn, Great Lakes Carbon Corp., private communication, April 24, 1973

TABLE XXX

PRODUCTION RUN 75 TO 100 μ (3 TO 4 MIL) DIAMETER FIBER

<u>Exp. #</u>	<u>Substrate Lot #</u>	<u>Length</u>		<u>Diameter</u>		<u>Ultimate Tensile Strength</u>	
		<u>Meters</u>	<u>Feet</u>	<u>μ</u>	<u>(mils)</u>	<u>N/cm² $\times 10^3$</u>	<u>psi $\times 10^3$</u>
P-647 ¹	1132	335	1100	89.7	3.53	212, 215, 286, 244, 241, 256, 207, 234, 221	307, 312, 415, 354, 350, 371, 300, 340, 320
P-648 ²	1132	335	1100	93.2	3.67	240, 232, 243, 236, 197, 234	348, 337, 352, 343, 286, 340
P-665 ²	1144	357	1170	90.2	3.55	216, 187, 234, 222, 208	313, 271, 340, 322, 301
P-669 ²	1149	276	905	90.2	3.55	217, 214, 207, 217, 189	314, 310, 300, 314, 274
P-671 ²	1144	177	580	94.2	3.71	248, 268, 255, 236, 217	360, 389, 370, 342, 315
Mean						228	330
Standard Deviation						22	32
Coefficient of Variation						.10	.10

.64 cm (1/4") gauge length¹ Modulus = 18.2 N/cm² $\times 10^6$ (26.4 $\times 10^6$ psi)² Modulus = 17.9 N/cm² $\times 10^6$ (26.0 $\times 10^6$ psi)

TABLE XXXI

PRODUCTION RUN 140μ (6 MIL) DIAMETER FIBER

Exp. #	Substrate Lot #	Length		Diameter		Ultimate Tensile Strength	
		Meters	Feet	μ	(mils)	$\text{N/cm}^2 \times 10^3$	$\text{psi} \times 10^3$
P-537	1132	183	600	143	5.63	174, 152, 248, 228, 254	253, 221, 359, 330, 368
P-538 ₁	1132	76	250	142	5.58	203, 199, 218, 171, 201	295, 288, 316, 248, 292
P-540	1102	61	200	142	5.59	268, 307, 330, 335, 302	388, 445, 478, 486, 438
P-538 ₂	1132	76	250	143	5.63	161, 276, 255, 203, 224	234, 400, 370, 295, 325
P-575	1132	69	225	141	5.56	305, 331, 261, 300, 290	443, 480, 378, 435, 420
P-577	1132	82	270	142	5.61	352, 357, 279, 341, 218	510, 518, 405, 495, 316
P-578	1132	101	330	144	5.66	241, 145, 328, 278, 331, 294	350, 210, 475, 403, 400, 426
P-580	1132	91	300	141	5.56	285, 165, 255, 207, 193, 279	413, 240, 370, 300, 280, 405
P-581	1132	91	300	142	5.61	262, 252, 194, 362, 279	380, 365, 282, 525, 405
P-582	1132	107	350	146	5.73	203, 265, 317, 262, 241	295, 384, 460, 380, 350
P-584	1132	94	310	145	5.69	195, 355, 369, 341, 214	283, 515, 535, 495, 310
Mean						261	378
Standard Deviation						60	87
Coefficient of Variation						.23	.23
2.54 cm (1 in) gauge length							
Modulus = $19.3 \text{ N/cm}^2 \times 10^6$ ($28 \times 10^6 \text{ psi}$)							